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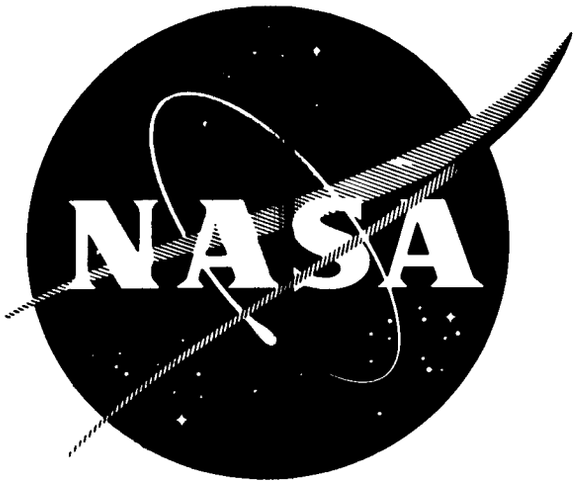

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

Quarterly Progress Report No. 3
For Quarter Ending April 15, 1964

EDITED BY E. E. HOFFMAN

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
Contract NAS 3-2547

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI 15, OHIO

POTASSIUM CORROSION TEST LOOP DEVELOPMENT

QUARTERLY PROGRESS REPORT 3

Covering the Period

January 15, 1964 through April 15, 1964

Edited by
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Project Manager

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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Under Contract NAS 3-2547

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SPACE POWER AND PROPULSION SECTION
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POTASSIUM CORROSION TEST LOOP DEVELOPMENT

I INTRODUCTION

This report covers the period, from January 15, 1964 to April 15, 1964, of a program to develop a prototype corrosion test loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The envisioned prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in a primary loop and will be used in a heat exchanger to boil potassium in the secondary, corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperatures could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F
2. Superheat temperature, 2000°F
3. Condensing temperature, 1350°F
4. Subcooling temperature, 800°F
5. Mass flow rate, 20 to 40 lb/hr
6. Vapor velocity, 100 to 150 ft/sec
7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr ft^2

The development program is proceeding with the construction and operation of three Cb-1Zr test loops, each of which will be used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260° to 2380°F to evaluate the electrical power vacuum feed-throughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase, forced-circulation loop, has been partially constructed and will be operated for 2,500 hours with liquid sodium at about 2100°F to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers. The Pre-prototype Corrosion Test Loop, a two-loop system, which is partially designed, will include a boiler, turbine simulator, and condenser in addition to the above components. This loop facility will be used to develop and endurance test (2,500 hours) the components required to achieve stable operation at the corrosion test design conditions.

The quarterly reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures, and test results. Detailed topical reports will also be issued to describe each test loop. Additional topical reports will be prepared to cover such areas as materials specifications, purification of potassium and sodium, and inert gas purification and analysis.

II PROGRAM STATUS

1. Component Evaluation Test Loop I

During the past quarter, Component Evaluation Test I successfully completed 1,000 hours of operation at an average maximum temperature of 2320°F. Loop I is a natural convection, liquid sodium loop constructed primarily of 0.375-inch OD by 0.065-inch wall Cb-1Zr tubing and operating in an ultra high vacuum chamber capable of 1×10^{-9} torr operation. The primary objectives of the test were to evaluate the electrical power feed-throughs, the method of attaching electrodes, the electrical resistivity of the heater segments and the thermal and electrical insulation. Loop operation was interrupted after 500 hours to replace thermocouples. The test was re-started and continued uninterrupted for an additional 500 hours of operation. The operational results of Loop I are summarized in Table I and Figure 1.

The flow rate in a natural convection loop is a function of the net difference in the average density of the fluid between the hot and cold legs of the loop, the height of the loop, and the flow resistance of the loop. The flow rate in the loop will, therefore, remain constant once thermal equilibrium has been achieved. The experimental flow rate was calculated by equating the expression for the heat rejection by radiation from a section of the cold leg to the heat loss by the sodium in the same section of the cold leg

$$Q_1 = \sigma \epsilon A(T_1^4 - T_2^4) = \text{Btu/hour radiated}$$

where σ = Stefan-Boltzmann constant = 0.1714×10^{-8} $\frac{\text{Btu}}{\text{hr-ft}^2\text{-}^\circ\text{R}^4}$

ϵ = Emittance of emitter, 0.5

A = Area of emitter, ft^2

T_1 = Absolute temperature of emitter, $^\circ R$

T_2 = Absolute temperature of receiver, $^\circ R$

$$Q_2 = W\, c\, \Delta T = \text{Btu/hr lost from sodium}$$

where W = Weight rate of flow, lbs/hr

c = Heat capacity, Btu/lb-$^\circ$F

ΔT = Sodium temperature drop, $^\circ F$

and solving for the flow rate. The calculated flow rate of 11.4 lbs/hr was then used to check the heat balance in the heater sections and in the overall thermal balance of the loop. The results of these calculations show that approximately 50% of the total heat input of 1.7 KW was utilized

TABLE I

LOOP I OPERATION RESULTS

TEST DURATION - 1,000 HOURS

DESIGN TEMPERATURE - 2200°F

	0-500 Hours	500-1,000 Hours
Flow Rate		11 lbs/hr
Sodium Temperature, Heater Inlet	1400°F	1350°F
Sodium Temperature, Heater Outlet	2285°F	2200°F
Sodium Temperature, Maximum	2380°F	2260°F
Current Through Heater Coils		690 amps
Potential Across Heater Coils		2.4 volts
Total Power Input		1.7 KW
Power Input to Sodium		0.9 KW
Heat Losses in Heater Coils		0.8 KW

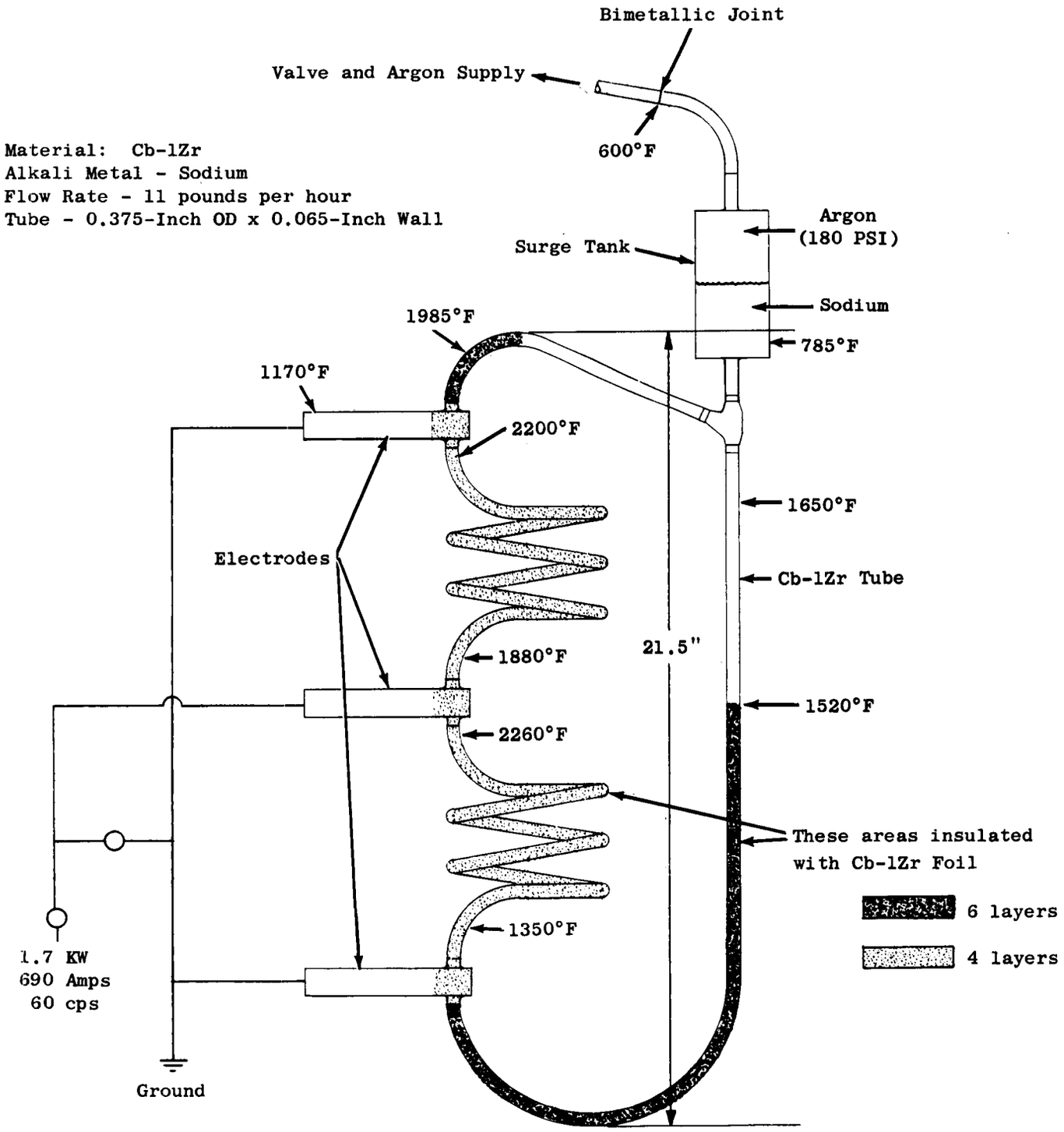

Figure 1. Component Evaluation Test Loop I. - (0800, 31 March 1964)

in increasing the inlet sodium temperature from 1350°F to an exit temperature of 2200°F. Heat losses from the electrodes and coils accounted for the remainder of the heat input. Future forced convection loop tests, where significantly greater flow rates will be required, will show a higher thermal efficiency since the heat losses will remain nearly the same as for Loop I but the heat input to the sodium will be increased by a factor of 10.

The electric resistance heater of the loop consists of two 4-inch diameter helical coils of 0.375-inch OD x 0.25-inch ID Cb-1Zr tube approximately 36 inches long with a common center electrode and two outer grounded electrodes. Current is conducted not only through the 0.065-inch tube wall but also through the flowing sodium. The heat generated in each path of a parallel circuit is inversely proportional to the ratio of the electrical resistance of each path and is also a function of the temperature of both the Cb-1Zr tube wall and the sodium. The heat generated in the lower section of the heater was approximately 20% higher than the heat generated in the upper coil due to its lower average electrical resistance. The result of the unbalance of power in the heater sections was that the highest temperature of the loop during the last 500 hours of the test (2260°F) was measured at the exit of the lower section. The sodium experienced a 380°F drop in temperature in passing through the heat sink created by the center 0.75-inch x 1-inch x 3.75-inch long heater electrode. The reduced power level of the upper section was only capable of restoring the temperature to 2200°F and to balance the radiation heat losses. The sodium experienced a 215°F temperature drop in passing through the top electrode which had only 1/2 the cross sectional area of the center electrode.

The temperature distribution in the top electrode is shown in Figure 2 and is in agreement with the calculated temperature distribution for a finite rectangular fin with no internal heat generation. The 61 BTU/hr of heat generated in the electrode by I^2R electrical losses amounted to only 8% of the 768 BTU/hr of heat radiated by the electrodes and was neglected in determining the calculated temperature distribution.

On the basis of the above measurements, the electrodes for future tests will be redesigned to reduce heat losses. The lower heat losses and the higher flow rates of the forced convection loops will permit more accurate thermal balances and provide a secondary calibration on the flow rate to be measured by a permanent magnet flowmeter.

The objectives of the Component Evaluation Test I to prove specific components to be used in succeeding tests of the Corrosion Loop Development Program were realized in all areas. The specific objectives and their evaluation are presented in Table II.

Prior to the loop operation, the entire vacuum chamber and loop were thoroughly baked out at 400°F. The chamber was cooled to room temperature and all flanges, feed-throughs and valves were helium leak checked using a

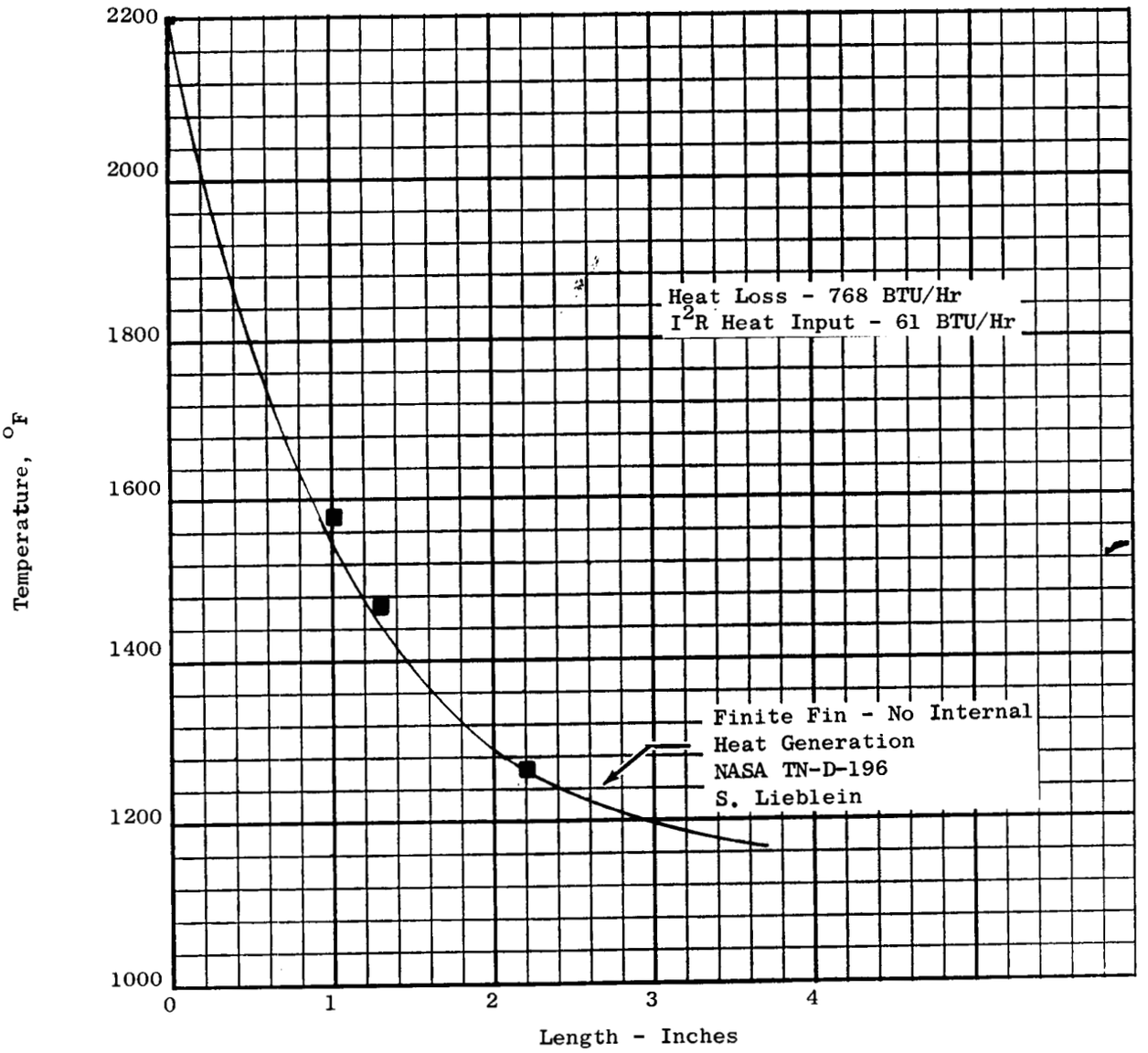

Figure 2. Temperature Distribution in Top Cb-1Zr Electrode (0.5-Inch x 0.75-Inch x 3.75-Inch Long) During Loop I Operation.

TABLE II

SUMMARY - COMPONENT EVALUATION TEST LOOP I

1. 1000-ampere vacuum feed-throughs	No deterioration of seal or leakage observed - maximum current of test - 690 amperes.
2. Cb-1Zr electrodes	Performance satisfactory - to be redesigned with a thermal barrier to reduce heat losses.
3. Electrical resistivity of heater	Resistance agreed within 20% of predicted values at test conditions.
4. Thermal insulation	Six layers of dimpled 0.005-inch thick Cb-1Zr foil proved an effective and convenient method of thermally shielding all components.
5. Electrical insulation	99.7% alumina insulators for both power leads and thermocouple leads presented no problems in either contamination or outgassing. The maximum insulator temperature was approximately 2300°F on several of the thermocouple insulators in the heater section.
6. Tungsten-rhenium thermocouples	Installation procedures were developed. The two major thermocouple problems encountered were: 1) breaking of the 0.005-inch diameter W-3Re leg of the thermocouple and 2) spurious emfs generated by intermittent contact of the thermocouple wire with the vacuum feed-through tubulation. Improved handling techniques were developed to eliminate fracturing of the wire. Reducing the length of the brazed thermocouple seal and insulating the thermocouple wire from the nickel feed-through tubulation eliminated the spurious emfs mentioned above.

G.E. Partial Pressure Gas Analyzer as the leak detector. Upon completion of the leak check, the tank bakeout heaters were turned on and the loop temperature increased to 250°F. Power to the loop heater was slowly increased so that the loop pressure would not exceed 1×10^{-6} torr. Approximately 6 hours were required before full power could be applied to the heater and the loop attain thermal equilibrium at a heater outlet maximum temperature of 2285°F.

During the first 500 hours of operation, the Partial Pressure Gas Analyzer was used to determine qualitatively the residual gases in the vacuum chamber. Although the Partial Pressure Gas Analyzer had not been calibrated for the absolute pressure of the residual gases, it had been previously established that the electrical output of the analyzer was a linear function of the absolute pressure, and changes in the partial pressure of the residual gases could be measured as a function of time. Figure 3 shows that the change in the partial pressure of the principal residual gases, H_2, CO and N_2 immediately started to decay, indicating that the principal constituent of the outgassing of the heated loop is hydrogen. Similar observations have been reported for other refractory metal systems heated in high vacuums. Upon cooling the loop after completion of the test, the observed pressure was 3.4×10^{-9} torr.

Following removal of the loop from the test chamber, the sodium will be drained from the loop. The various sections of the loop will be examined carefully to determine if any mass transfer deposits are present. Metallographic specimens will be taken from several regions and the hottest unwrapped portion of the loop will be chemically analyzed for the interstitial elements.

2. Component Evaluation Test Loop II Design

The design of Loop II, the single-phase, forced convection loop in which sodium will be circulated at 2100°F was completed and approved for fabrication. The principal features of the loop and an isometric drawing of the system were included in the last progress report[1].

The 24-inch x 54-inch high vacuum test chamber from Varian Associates has successfully completed its acceptance test at General Electric by achieving a pressure of 1×10^{-9} torr in less than 24 hours without the use of the titanium sublimation pumps which are to be used in handling peak outgassing loads. The system reached a pressure of 2.4×10^{-10} torr in 48 hours and 1.5×10^{-10} torr after four days of pumping.

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[1] Potassium Corrosion Test Loop Development, Quarterly Progress Report 2, Covering the Period October 15, 1963 through January 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54008.

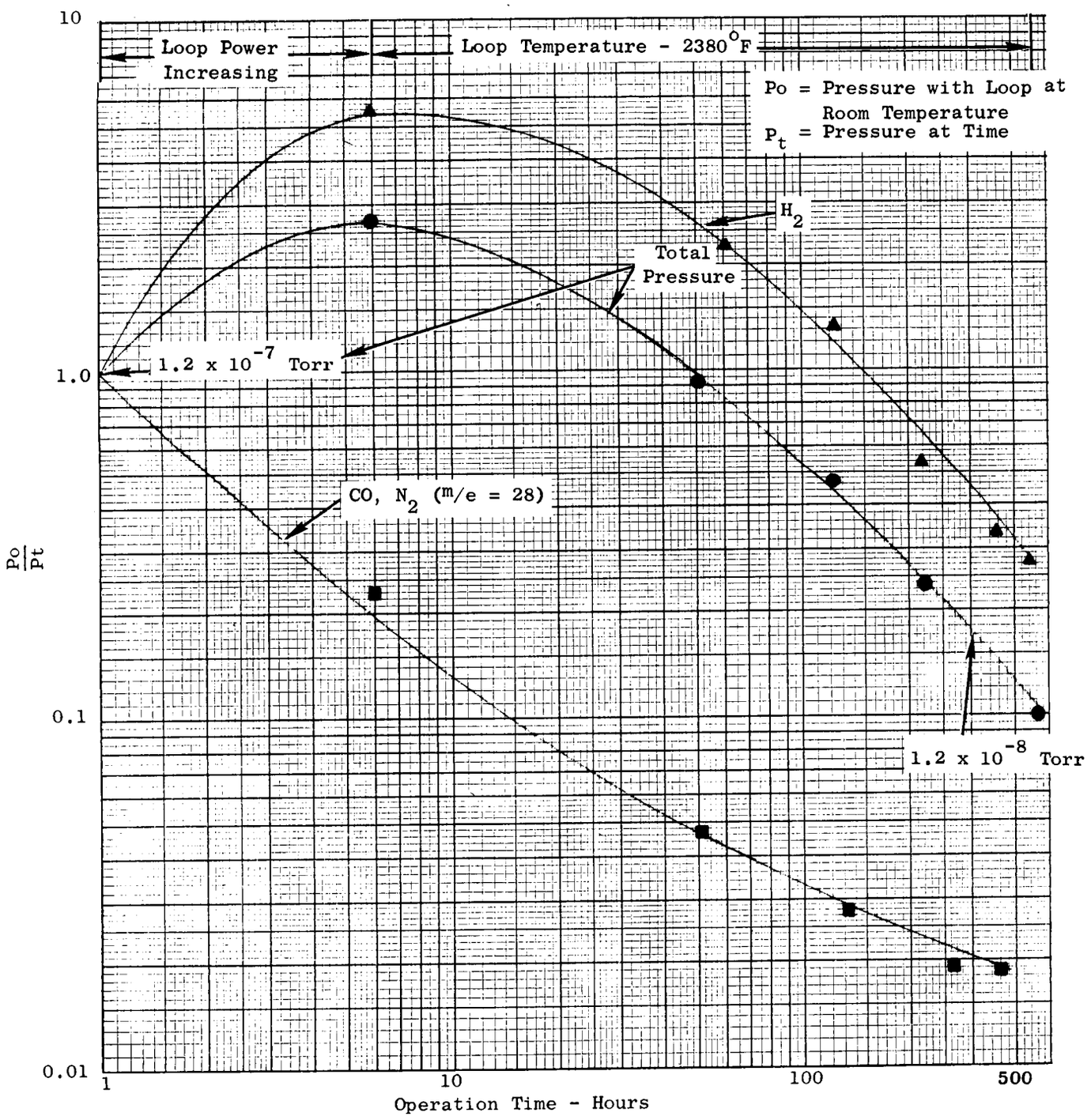

Figure 3. Variation of Total and Partial Pressures During First 500 Hours of Loop I Operation.

3. Loop II Fabrication

The welding of seven Cb-1Zr bellows valves to be used on Loop II and the Pre-prototype Loop has been completed. The sequence of manufacturing operations was bellows fabrication (Standard-Thomson, Inc.), welding of the bellows assembly (General Electric), final machining and fit-up of plugs and valve bodies (Hoke, Inc.), and the welding of the bellows assembly to the valve body (General Electric). Tests of valve performance, flow control, shut-off torque, etc., are currently being conducted to supplement those reported by Hoke, Inc.

The interstitial element contents of the bellows blanks before and during fabrication are compiled in Table III and include both vendor data and results of General Electric testing.

The oxygen concentration, although higher than desired, is considered representative of available thin-walled tube technology. Further vendor liaison is indicated to improve the quality of both as-received tube and vacuum heat treating facilities.

The bellows fabrication by Standard-Thomson, Inc. was done in two forming operations. The first forming operation produced the deformation illustrated in Figure 4. After intermediate heat treatment, at 2300°F for one hour, the second, more severe, forming was attempted. Initial trials produced many failures such as those shown in Figure 5. Reduction of the hydraulic pressure and adjustment of other forming variables resulted in the production of high quality bellows, as indicated by the bellows cross section illustrated in Figure 6. The maximum thickness variation was approximately ten per cent. For comparison, Cb-1Zr bellows fabricated by another vendor had thickness variations of about 100%, as shown in Figure 7.

A total of 94 Cb-1Zr bellows were processed to assure that a minimum of 10 good bellows were obtained for making the required valves. Three processing paths were followed and the results are summarized in Table IV.

Seven completed bellows were delivered to Hoke, Inc. for fitting to the bellows assembly. These assemblies were electron beam welded at General Electric and returned to Hoke, Inc. for final machining and fitting of the valve plugs. The completed bellows assembly and plug are shown in Figure 8. The appearance of the bellows is typical.

The component parts of the valve are shown in Figure 9. Final fabrication involved only a seal weld between the bellows assembly and the valve body. To avoid any possible distortion of the valve seat, the electron beam welding process was utilized. The finished valve without the stainless steel components is shown in Figure 10.

TABLE III

RESULTS OF CHEMICAL ANALYSES BEFORE AND DURING BELLOWS FABRICATION

		Chemical Analysis, ppm			
		C	O	N	H
1.	Ingot Analysis (Superior Tube)	110	250	60	3
2.	Target Analysis*	200 (max)	500 (max)	300 (max)	50 (max)
3.	Tube, As-Shipped** (Superior Tube)	210	660	72	9
4.	Tube, As-Received (General Electric)	190, 220	868, 880	102, 104	7, 13
5.	Tube After 1st Vacuum Heat Treatment, 1 Hour-2300°F (Stellite Division, UCC)	140, 160	826, 828	106	1, 2
6.	Tube After 1st Forming and Second Heat Treatment, 1 Hour-2300°F (Stellite Division, UCC)	--	1180, 1230	119, 87	1, 1

* Target analysis based on vendor experience. Material procured on a "best efforts" basis.

** Tube size: 0.375-inch OD x 0.008-inch wall.

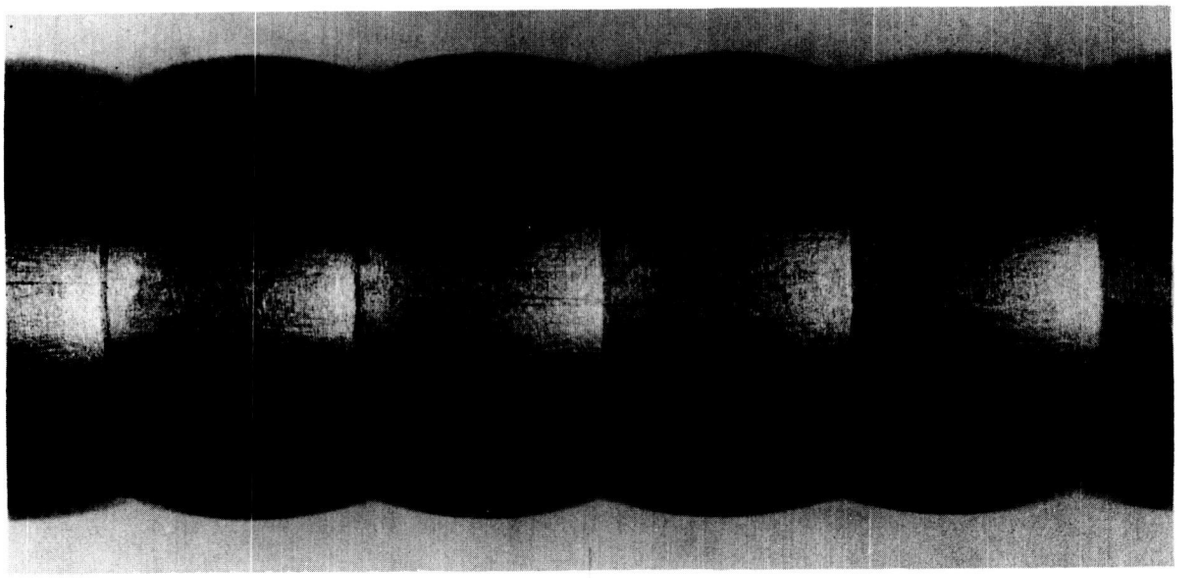

Figure 4. Cb-1Zr Bellows Following First Stage of Fabrication at Standard-Thomson, Inc.

Starting Tube Blank Dimensions: 0.375-Inch OD x 0.008-Inch Wall x 7-Inch Long.

Mag: 7X (C63122038)

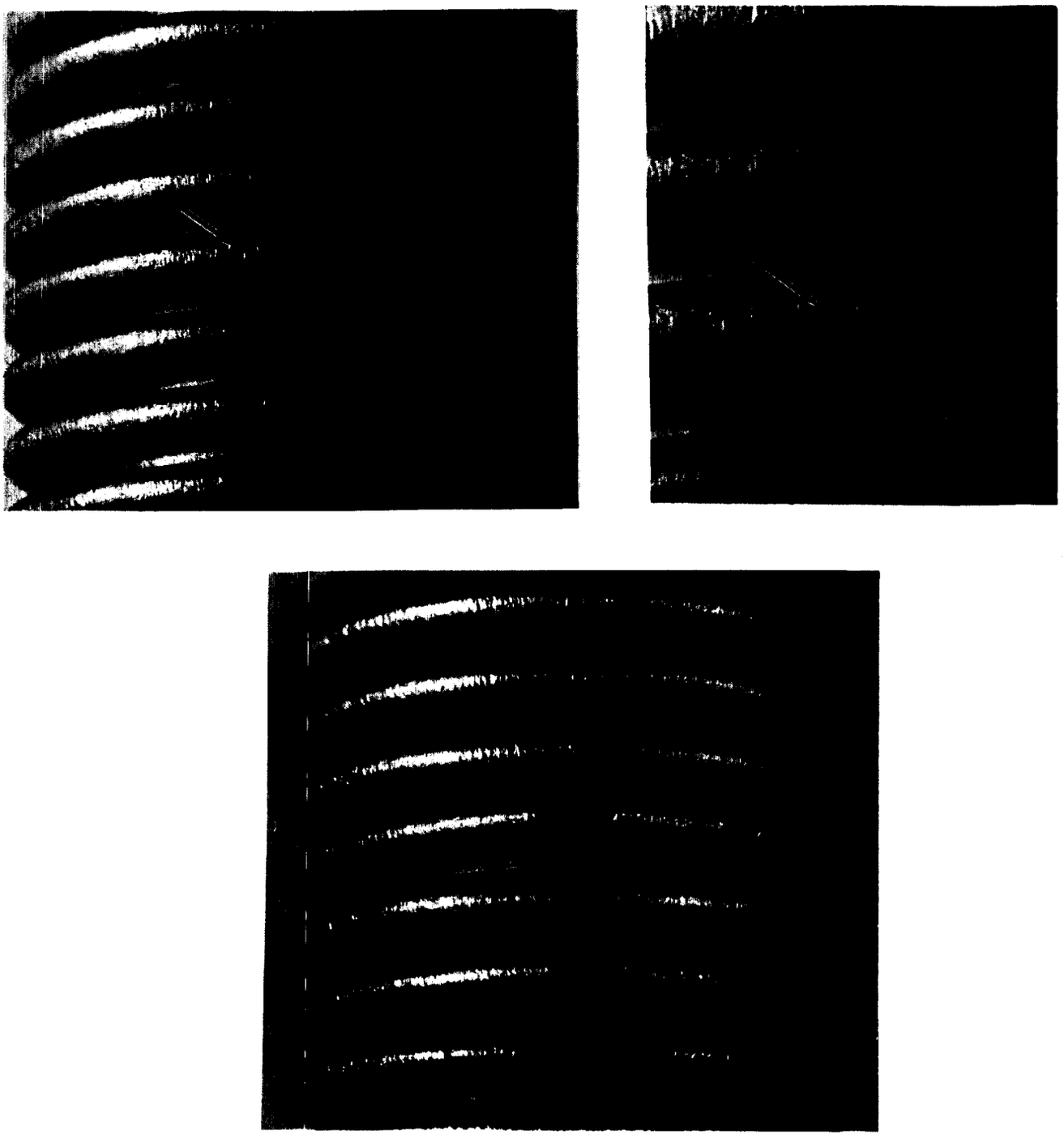

Figure 5. Cb-1Zr Bellows Following Initial Trials of Second Stage Fabrication at Standard-Thomson, Inc.

Top - Left: Typical Superficial Defect Mag: 8X (C64010801)
Right: Enlarged View of this Defect Mag: 16X (C64010802)

Bottom - Complete Failure of Bellows Mag: 8X (C64010803)

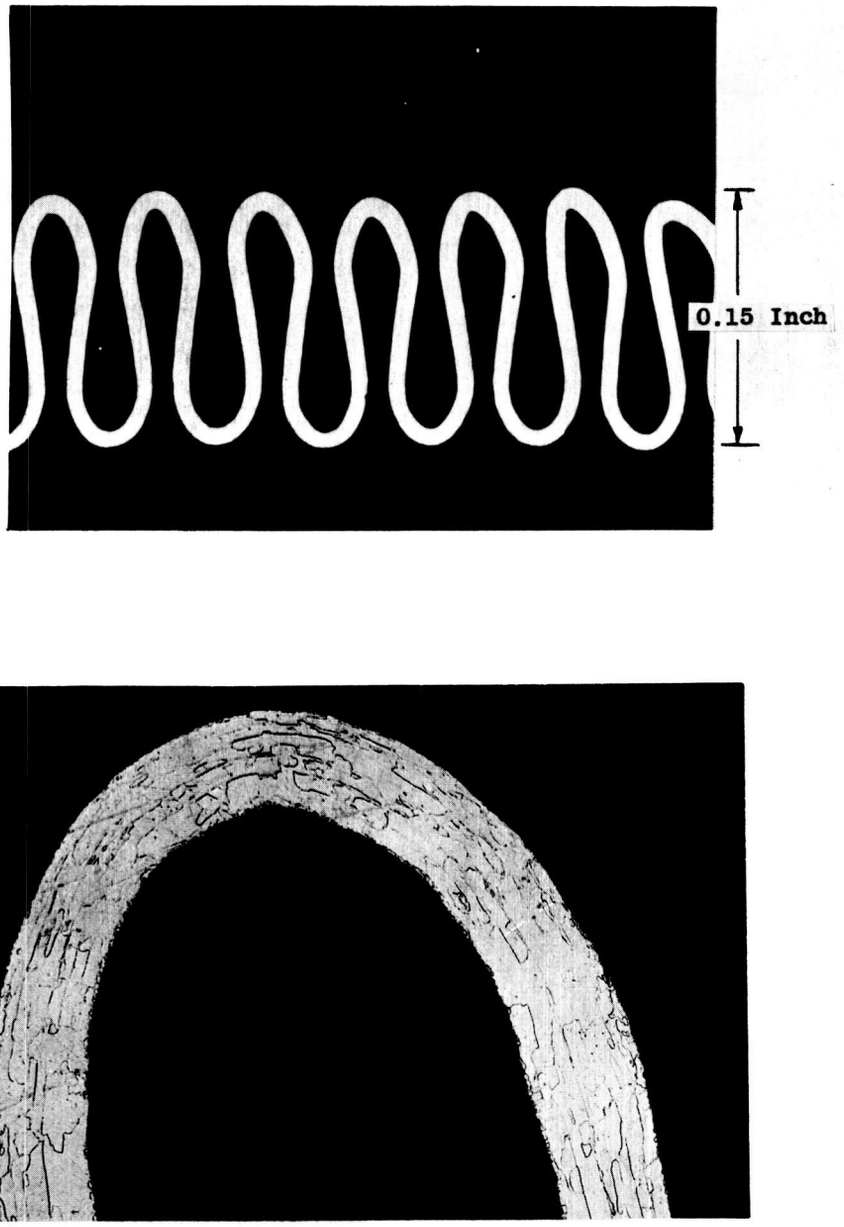

Figure 6. Cross Section of Cb-1Zr Bellows After Forming at Standard-Thomson, Inc.
Top - As-Polished Mag: 10X (C64022123)
Bottom - Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 100X (K249)

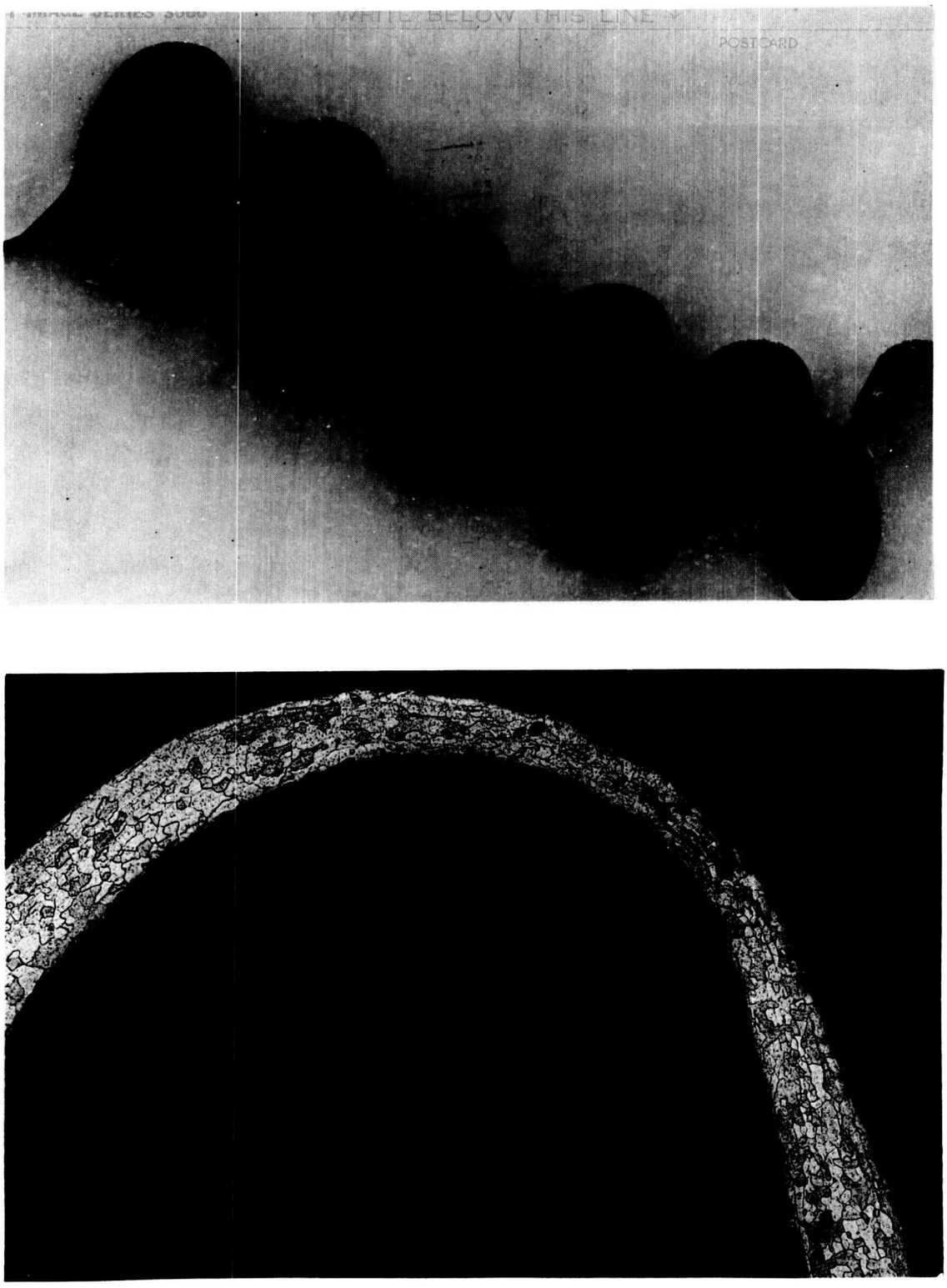

Figure 7. Cross Section of Cb-1Zr Bellows After Forming by Another Vendor.
Top - Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 10X (C2031620)
Bottom - Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 100X (Y1058)

TABLE IV

FABRICATION RESULTS OF Cb-1Zr BELLOWS

	Bellows History	Results
1.	a) Tube blanks annealed 1 hour-2300°F b) Initial forming operation at S-T* c) Annealed 1 hour-2300°F d) Final forming operation at S-T	Thirty-five of seventy-one bellows were tested and found to be helium leak tight by both S-T and G.E. Twenty of the thirty-five bellows were determined to be free of major surface defects as determined by microscopic examination at 30X.
2.	a) Tube blanks annealed 1 hour-2300°F b) Initial forming operation at S-T c) Final forming operation at S-T	Two of seven bellows were helium leak tight.
3.	a) Tube blanks annealed 1 hour-2300°F b) Final forming operation at S-T	Three of sixteen bellows were helium leak tight.

*Standard-Thomson, Incorporated

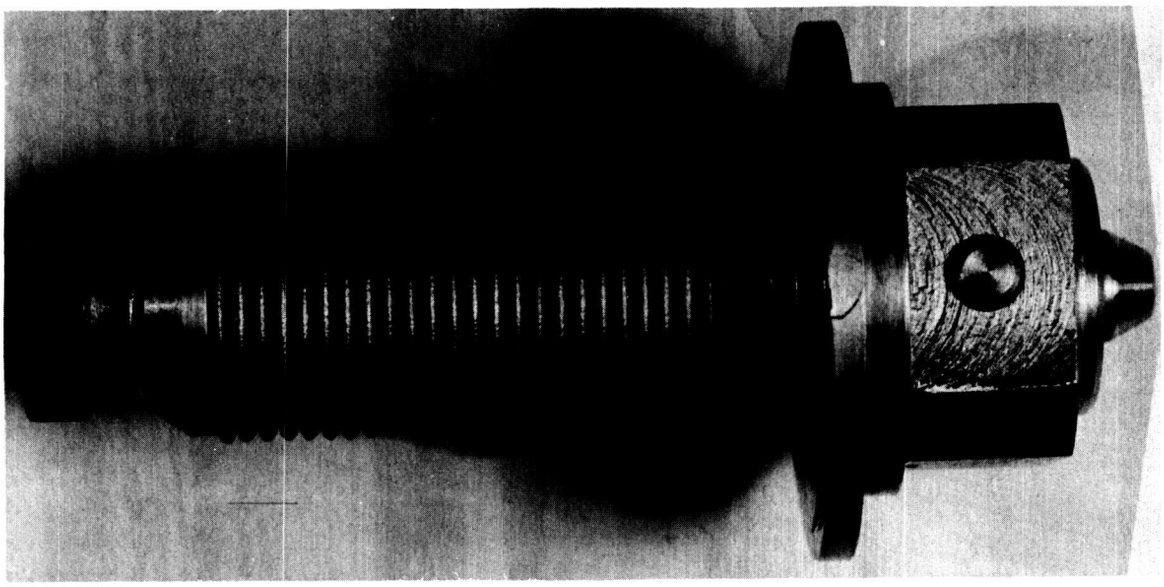

Figure 8. Bellows (Cb-1Zr) and Plug (Mo-TZM) Assembly of Hoke Valve for Loop II.

Mag: 3X (C64040908)

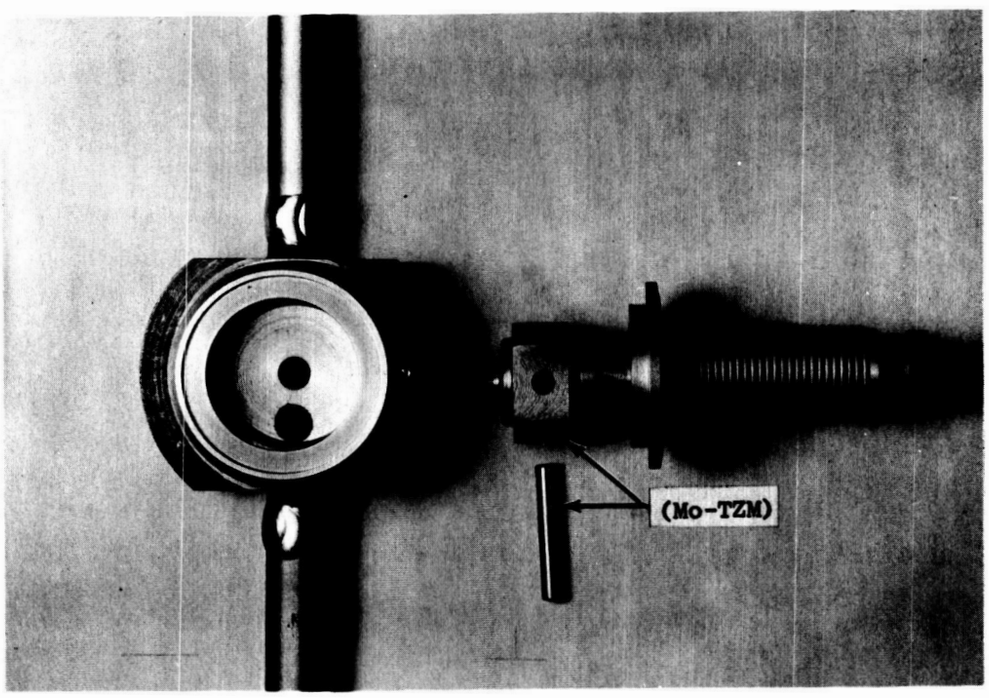

Figure 9. Component Parts of Hoke Bellows Valve Prior to the Final Welding Operation.

Mag: 1X (C64040205)

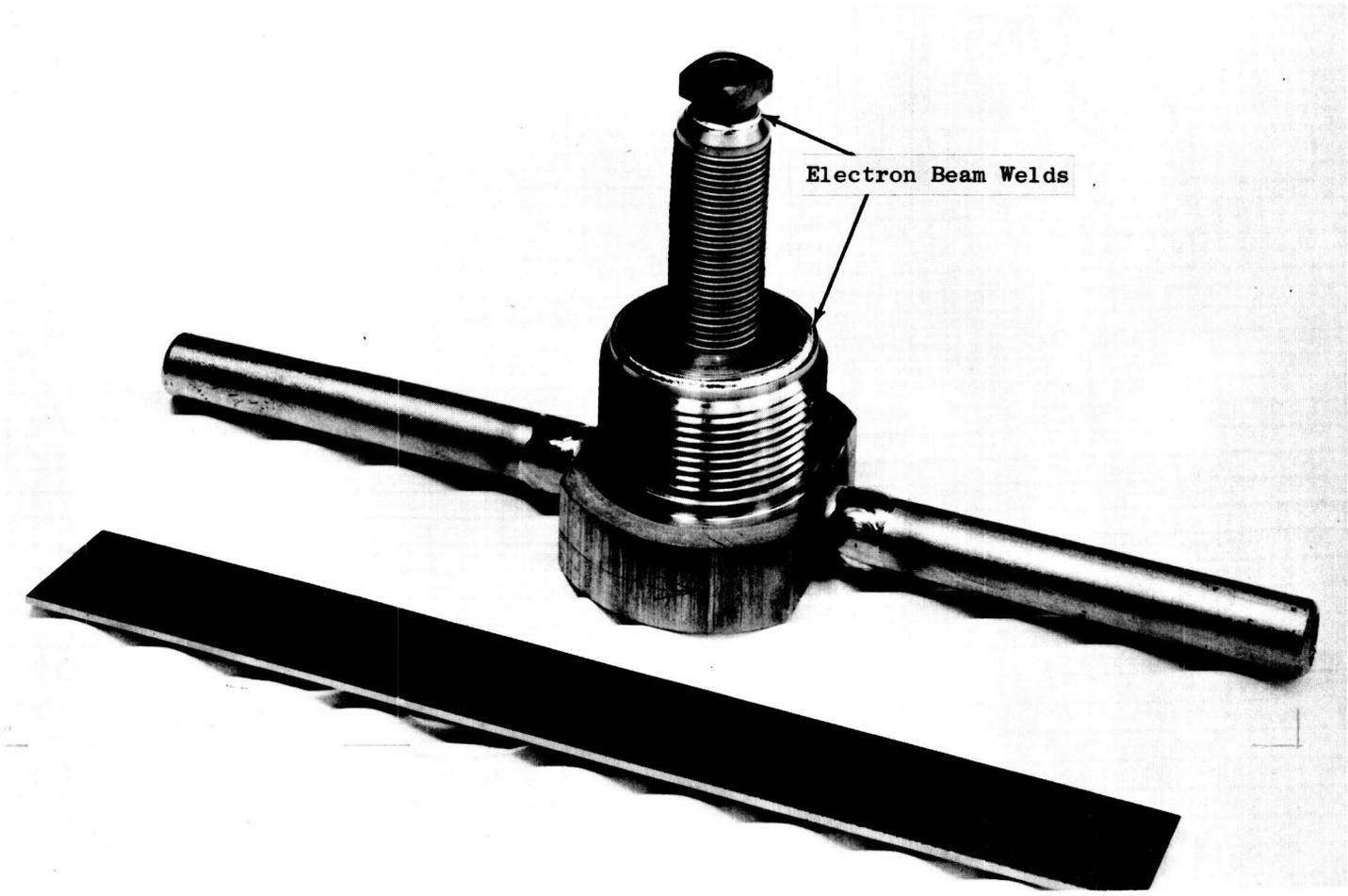

Figure 10. Cb-1Zr Hoke Bellows Valve After Completion of Welding Operations. (C64041328)

The fabrication of eight Taylor Instrument Company pressure transducers to be used on Loop II and the Pre-prototype Loop has been completed. These transducers have been shipped to the vendor for filling of the pressure transmitting capillary with NaK. Two transducers, one of which will be used on Loop II, will be filled with commercial purity NaK. A considerable program delay would have resulted if high-purity NaK had been specified for the first two transducers. Special high-purity helium has been supplied to Taylor by General Electric for use during the filling operation. The remaining six transducers for the Pre-prototype Loop will be filled at a later date with high-purity NaK.

A sample of the NaK used to fill the transducers will be taken for both sets of transducers at the time of filling. The specimen tubes, one of which is shown in Figure 11, will be processed and filled in the same manner as the pressure transducers. An apparatus capable of handling liquid alkali metal samples for the amalgamation method for oxygen has been designed, built and used to analyze NaK. Three analyses on 10 lbs of high-purity eutectic NaK from MSA Research Corporation produced values of 7.5, 11.1 and 5.6 ppm O. The same type of high-purity NaK will be used by Taylor for filling the second set of transducers.

The weld between the diaphragm and upper flange was made by the electron beam process. Tungsten, inert gas welding was used to join the process tube to the lower flange, the bimetallic joint assembly to the upper flange, and the final weld between upper and lower flanges. These components are illustrated in Figure 12. The trial electron beam weld is shown on the left and the completed upper flange on the right of Figure 12. The cross section of the electron beam diaphragm weld is shown in Figure 13. Radiographic inspection, heat treatment, and mass spectrometer leak tests were completed successfully before shipment to Taylor Instrument Company.

The electron beam welding of the above components was performed in accordance with Specification SPPS-14. Chemical analyses of the weld metal before and after each sequence of welds are reported in Table V. The oxygen, nitrogen and hydrogen contents indicated no contamination. The carbon content exceeded specification, i.e., less than 10 ppm increase, for the weld sample prepared before welding of the Hoke valve bellows assembly to the valve body. Because of the small sample size necessitated by the relatively narrow electron beam weld, these values are somewhat questionable. Duplicate analyses of the same sample indicated variations greater than plus or minus 10 ppm, which was the analytical variance anticipated per Specification SPPS-14.

The EM pump duct was machined, dye penetrant inspected, and the outer duct was ultrasonically inspected successfully. The process tubes were welded to the end caps prior to the production of the interference fit between the finned duct and outer duct. The EM pump duct components are shown in Figure 14.

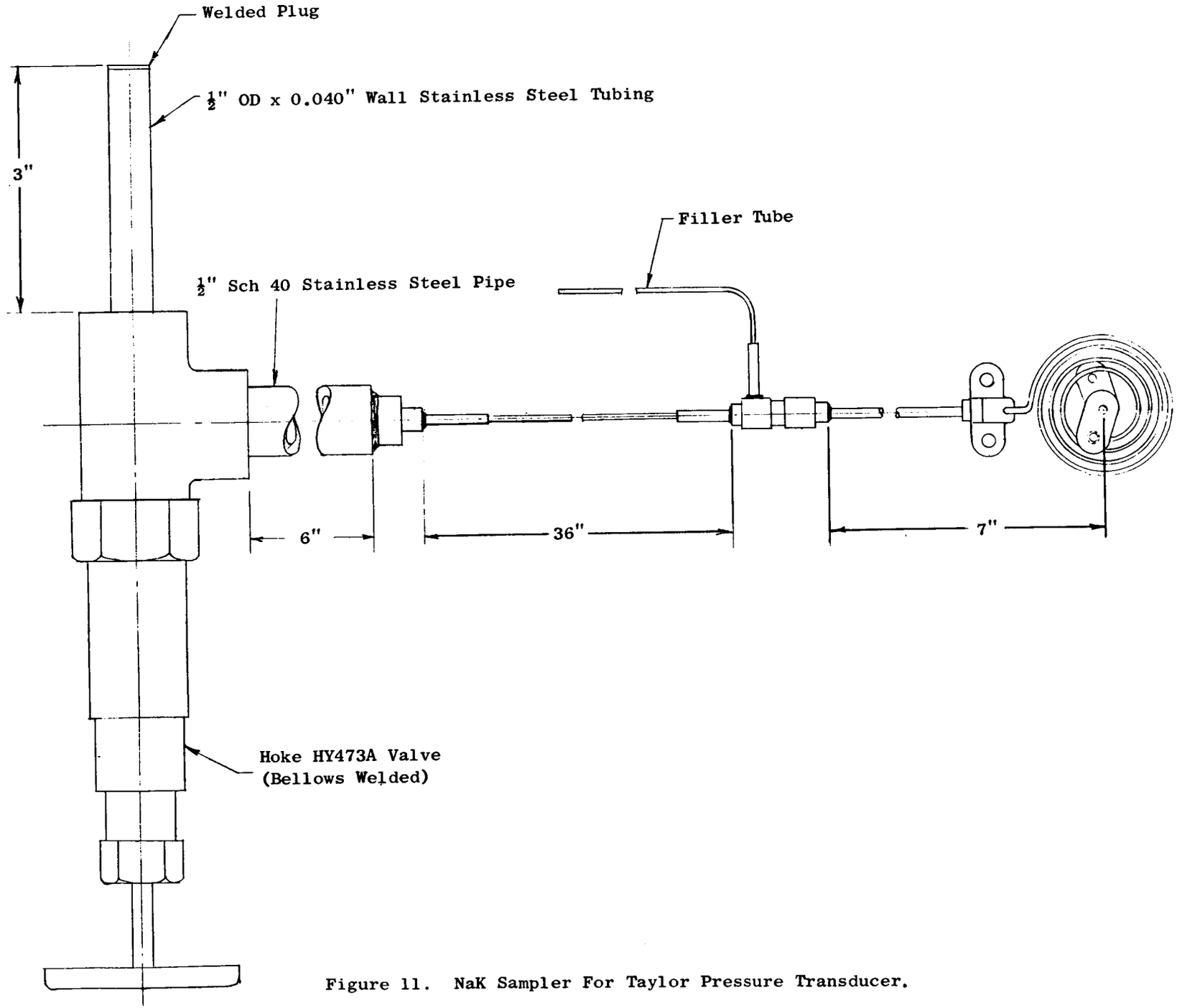

Figure 11. NaK Sampler For Taylor Pressure Transducer.

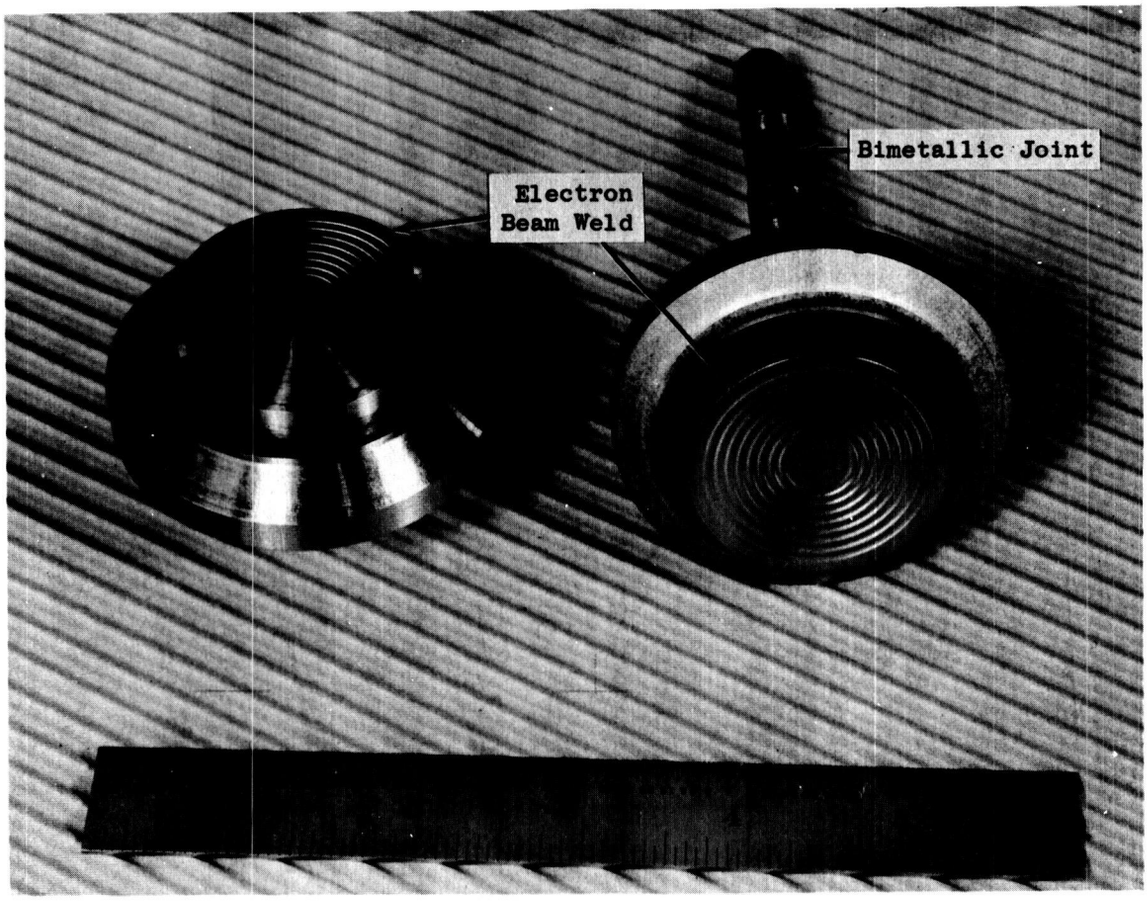

Figure 12. Taylor Cb-1Zr Pressure Transducer Upper Flange Assembly.
Left: Trial Electron Beam Weld
Right: Completed Upper Flange with Diaphragm and Bimetallic Joint Welded in Position

(C64021713)

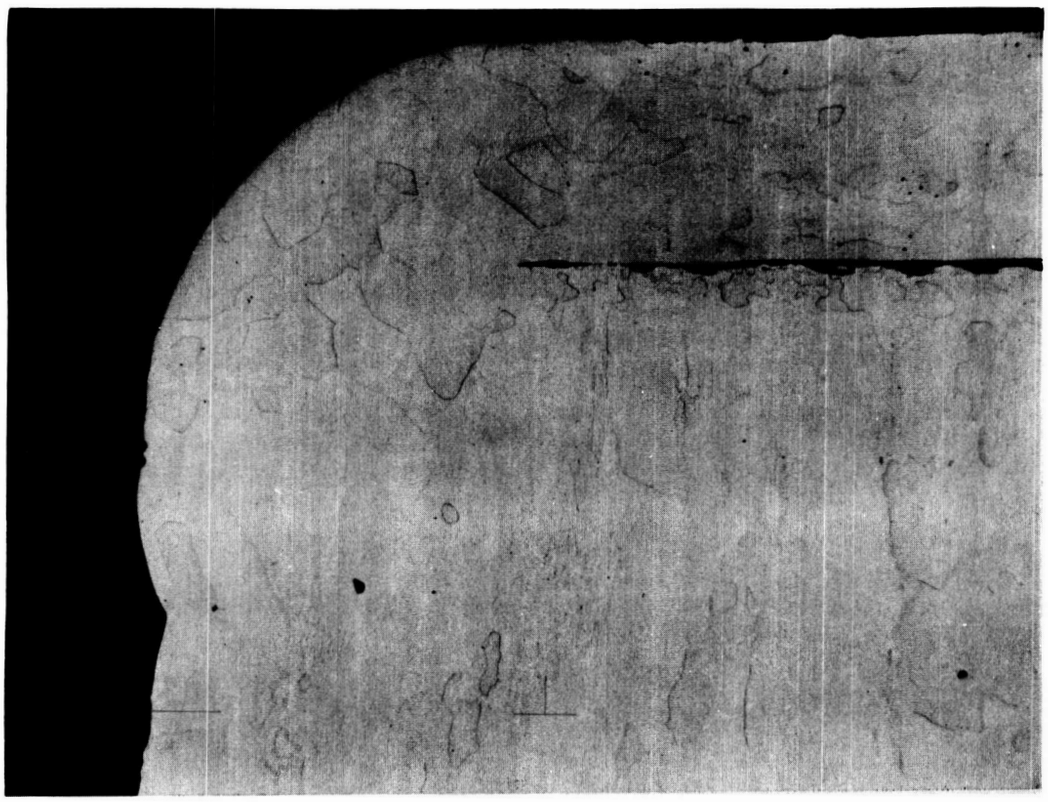

Figure 13. Cross Section of Electron Beam Weld Between Cb-1Zr Diaphragm and Cb-1Zr Upper Flange of Taylor Pressure Transducer.

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 250X (K492)

TABLE V

RESULTS OF CHEMICAL ANALYSES OF ELECTRON BEAM WELDMENTS PER SPECIFICATION SPPS-14

	Chemical Analysis, ppm			
	O	N	H	C
Base Metal Analysis by Vendor	130	85	1	50
Base Metal Analysis by General Electric	142	22	2	30, 40
Hoke Valve Bellows Assembly Welds				
Sample Weld[a] Before[b]	94	53	<1	100, 70
Sample Weld After	104	55	<1	40, 60
Hoke Valve Bellows Assembly to Body Weld				
Sample Weld Before	96	56	3	40, 70
Sample Weld After	110	57	3	0, 20
Taylor Pressure Transducer Diaphragm to Upper Flange Weld				
Sample Weld Before	107	43	2	10, 20
Sample Weld After	Not Determined			

(a) All weld metal analyses are on welds made on the base material listed above, before and after welding of components listed.

(b) All welds made at a pressure of $< 5 \times 10^{-5}$ torr.

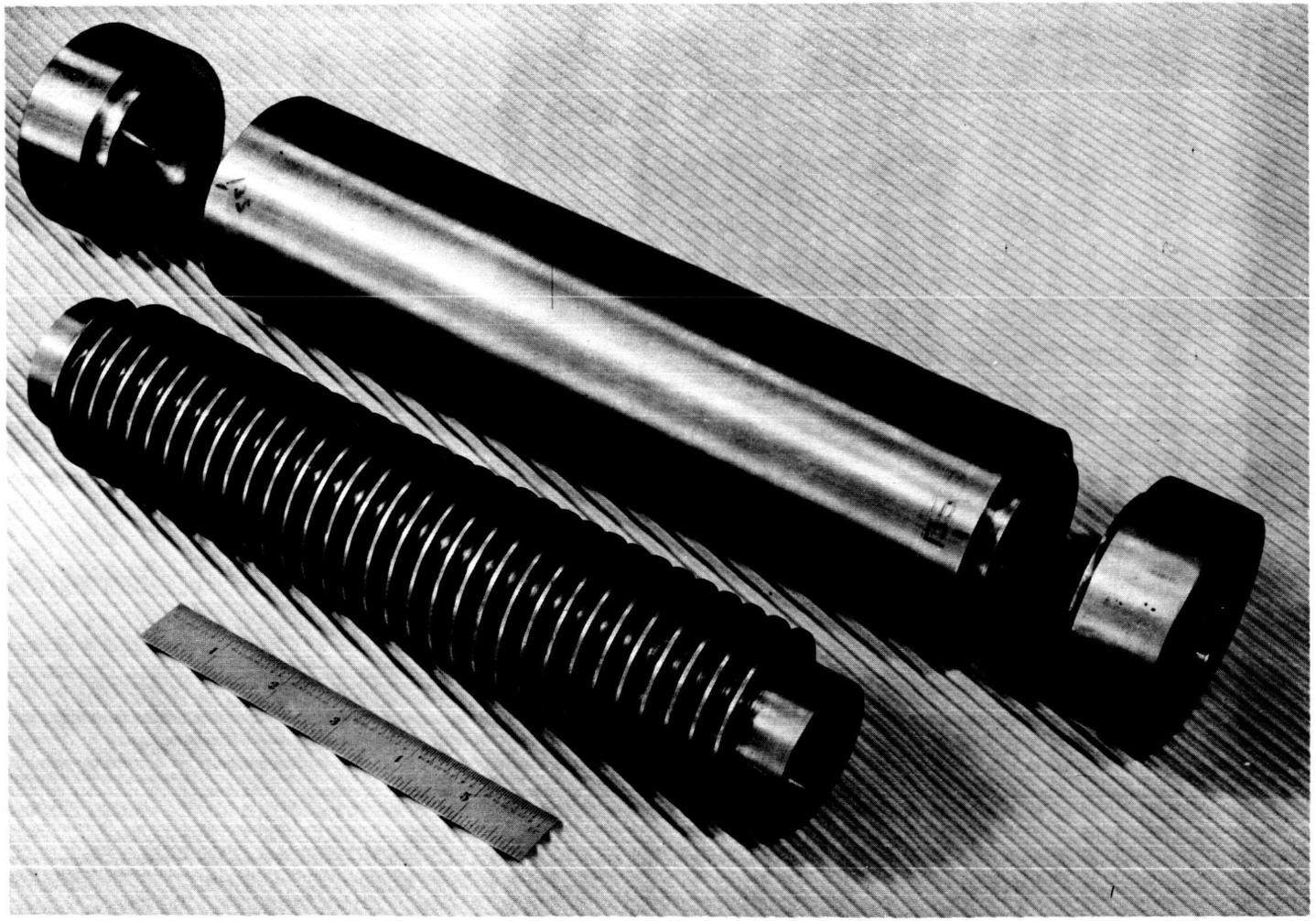

Figure 14. Cb-1Zr Alloy Pump Duct Components of Helical Induction Electromagnetic Sodium Pump for Component Evaluation Test Loop II.

(C64021714)

Additional progress on Loop II fabrication included receipt of the support structure, completion of welding of the surge tank, and completion of process tube forming operations.

4. Sodium for Loop II

Loop II will be filled with reactor grade sodium, purchased in accordance with specification SPPS-45-I, which has been outgassed and hot trapped according to specification SPPS-45-II. Filling of the loop will be accomplished by a measured volume technique. The all stainless steel equipment required for transfer and sampling is shown schematically in Figure 15.

The system has been designed with the following operation requirements in mind:

A. Bakeout the system at 300°F and obtain a system pressure of less than 1 x 10^{-4} torr prior to filling with sodium.

B. Flush all components which will be contacted by sodium with a "cleaning" charge of sodium.

C. Sample the sodium by means of a replaceable in-line sampler:

   i) while flushing the stainless steel fill system,

   ii) after circulating the sodium through the loop for 1 hour at 500°F,

   and

   iii) after completion of 2,500 hours of loop operation.

D. Dump the sodium from the loop into a disposal tank when required.

As may be seen by studying the schematic, the operation flexibility required has necessitated designing a rather complex plumbing system.

The 15-pound sodium hot trap which will be used is shown in Figure 16. This is an exploded view of the system components prior to making the final top closure weld. Sufficient zirconium sheet was used to obtain an alkali metal weight to getter surface area ratio of 3 grams/in^2. Sodium for the loop will be hot trapped at a temperature between 1350° and 1400°F for at least 100 hours.

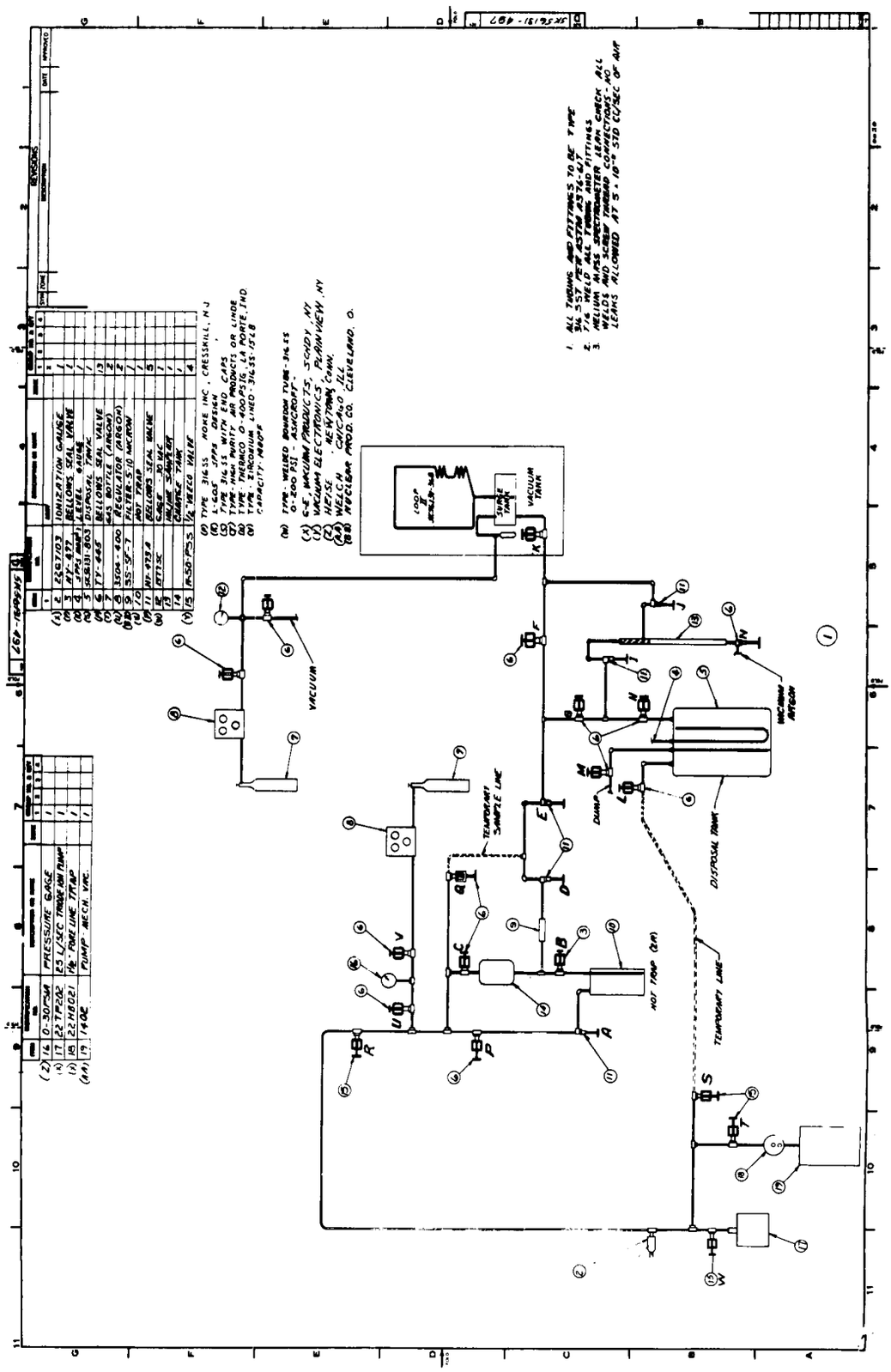

Figure 15. Schematic Drawing of Sodium Handling System for Loop II.

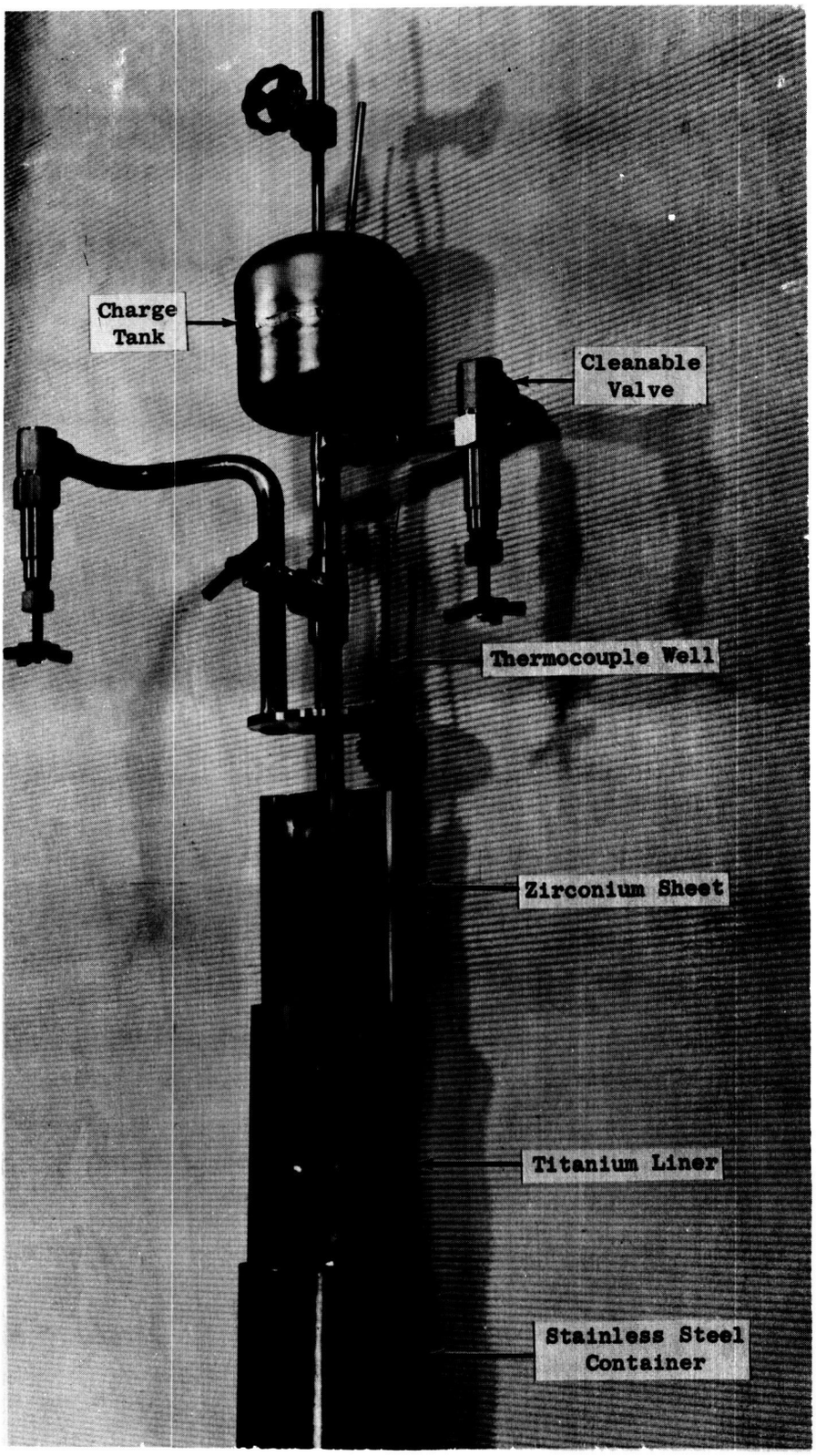

Figure 16. Sodium Hot Trap Tank and Charge Tank for Loop II. (C64041327)

5. Pre-prototype Loop Design

The detail design of the Pre-prototype Corrosion Test Loop has continued during the past quarter. Figure 17 is an isometric drawing of the Pre-prototype Loop and shows the relative position and orientation of the principal loop components which are briefly described below.

Primary Circuit

1. Electromagnetic pump rated at 5 gpm of sodium at 100 psi developed head and a temperature of 1900°F.

2. A 10 KW electrical resistance heater capable of exit sodium temperature of 2200°F.

Secondary Circuit

1. Electromagnetic pump rated at 40 pounds per hour of potassium at a developed head of 200 psi and a maximum potassium temperature of 1200°F.

2. 3/8-inch, Cb-1Zr bellows-seal metering valve to regulate the flow of liquid potassium.

3. 5 KW electrical resistance preheater capable of preheating the liquid potassium from 800°F to the boiler saturation temperature.

4. Tube-in-tube, counterflow boiler capable of converting 40 pounds per hour of liquid potassium to 100% quality vapor at 1900°F plus 100°F superheat.

5. Turbine simulator divided into two sections--the first section has one nozzle and blade pair and operates in the superheat region; the second section contains nine nozzle and blade pairs and operates in the 88% quality region. The vapor velocity at the throat of all nozzles is 1,000 feet per second.

6. Radiation-type tube condenser capable of rejecting approximately 10 KW of heat at 1350°F and subcooling the liquid to 800°F.

The entire loop is contained in a 48-inch diameter vacuum chamber capable of operating in the 10^{-9} torr region. During peak outgassing loads, 4 titanium sublimation pumps will assist in maintaining a pressure of less than 1×10^{-8} torr. The entire loop is supported by a removable center spool section to facilitate the fabrication and installation of the loop.

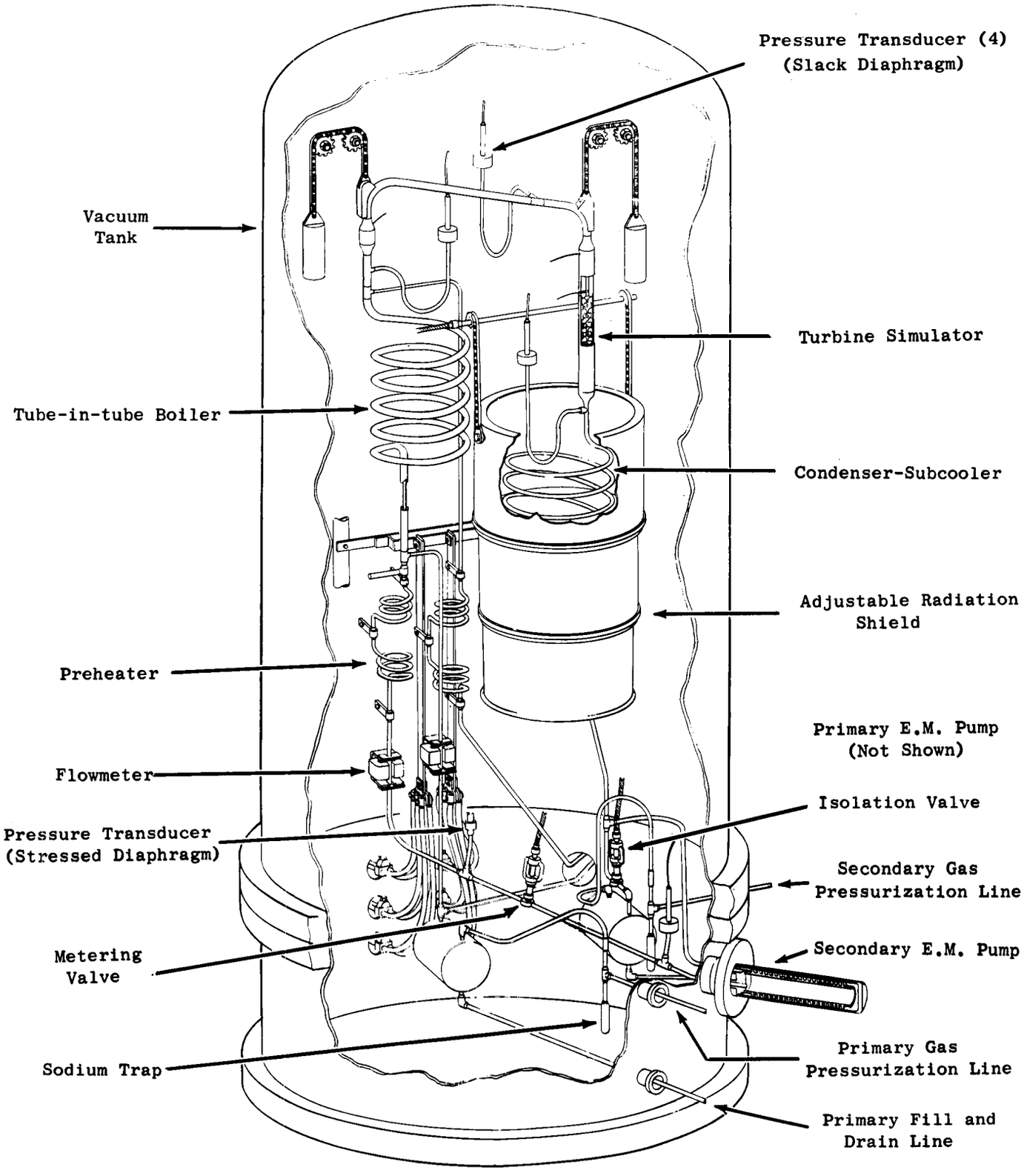

Figure 17. Isometric of the Pre-prototype Corrosion Test Loop.

6. Air Flow Pressure Drop Tests on Boiler Inserts

A series of air flow tests were completed to determine the flow characteristics of a flow swirling device or insert proposed for the Pre-prototype Loop boiler to promote heat transfer in the low quality (0-15%) entrance region. The flow swirler or insert consists of a 0.060-inch diameter wire helically wound around a 0.125-inch diameter center rod which is 12 inches long. The flow tests were made primarily to determine the effect of pitch on the pressure drop. The pitch is defined as the linear axial travel of the insert per revolution.

The 1/2-inch, 1-inch and 2-inch pitch inserts tested are shown in Figure 18; tests were also made for the tube without an insert for comparison with published friction factor data. A summary of the test conditions for the Pre-prototype boiler and the pressure drop test is given in Table VI. Typical tests data for the air flow tests are presented in Table VII. The test results are summarized in Table VIII for a tube Reynolds number of 40,000. The pressure drop for the 1/2-inch pitch was considered excessive and the 1-inch pitch insert was selected for the Pre-prototype boiler.

7. Pre-prototype Loop Fabrication

Six Taylor Instrument Company pressure transducers discussed under Loop II fabrication have been fabricated and returned to the vendor for filling with high-purity NaK. The Cb-1Zr bellows valves required for the Pre-prototype Loop have been completed.

The machining of the primary and secondary EM pump ducts is proceeding.

The boiler prototype which is being formed from half-hard copper has been delayed by the forming vendor. Completion is scheduled for early in the next report period.

8. Pre-prototype Loop Test Chamber

The 48-inch diameter by 128-inch high vacuum environmental test chamber for the Pre-prototype Loop was received and installed during the past quarter. The chamber and its auxiliary components are shown in Figure 19. The vacuum system had previously passed a preliminary acceptance test at the vendor's plant by achieving a pressure of 7.7×10^{-10} torr after a 24-hour bakeout and a 2.5 hours of additional pumping with the system cold. A pressure of 7.5×10^{-11} torr was reached in 2.5 hours after the titanium sublimation pumps were turned on.

Numerous attempts to repeat the above performance at General Electric by the vendor's field engineers have failed due to a sudden rise in pressure during the bakeout cycle. During these excursions, the pressure

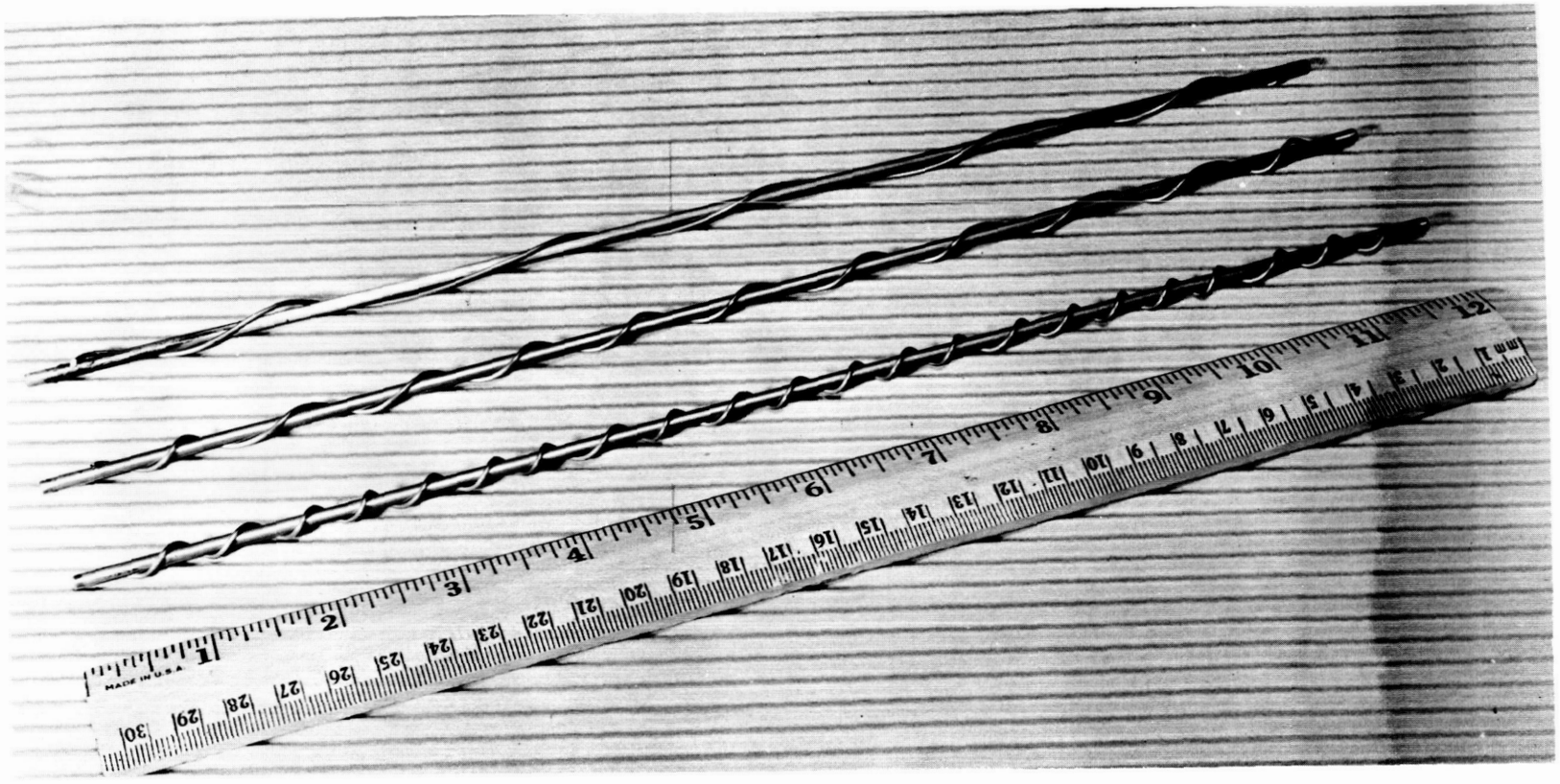

Figure 18. Boiler Tube Inserts Used in Pressure Drop Tests. (C64022107)

TABLE VI

SUMMARY OF PRE-PROTOTYPE DESIGN CONDITIONS AND

PRESSURE DROP TEST OF A BOILER INSERT

	Pre-prototype Loop	Pressure Drop Test
Gas	Potassium Vapor	Air
Temperature	1900°F	R.T.
Pressure at Exit	110 psia	14.7 psia
Viscosity	0.0535 lb/hr ft	0.045 lb/hr ft
Density	0.2 lb/ft^3	0.076 lb/ft^3
Tube ID	0.25 inch	0.25 inch
Maximum Flow Rate	40 lb/hr	33.5 lb/hr
Reynolds Number	46,700	46,700

TABLE VII

TESTS RESULTS OF 1/2", 1" AND 2" PITCH INSERT IN A 0.25" ID TUBE

Run No.	Flow Rate lb/Sec	Inlet Temp., °F	No Insert psi	2" Pitch psi	1" Pitch psi	1/2" Pitch psi
1	0.00104	69	0.034	0.265	0.435	1.33
2	0.00269	69	0.114	0.810	1.34	3.83
3	0.00311	69	0.226	1.60	2.55	7.15
4	0.00414	69	0.372	2.47	4.17	16.69
5	0.00706	69	0.916	6.22	9.37	23.3

TABLE VIII

FRICTIONAL PRESSURE DROP CHARACTERISTICS OF THE PRE-PROTOTYPE BOILER INSERT IN AIR AT Re = 40,000

	Friction Factor	Friction Factor Multiplier
Bare Tube - No Insert	0.0067	1
2-Inch Pitch Insert	0.048	7
1-Inch Pitch Insert	0.075	11
1/2-Inch Pitch Insert	0.255	38

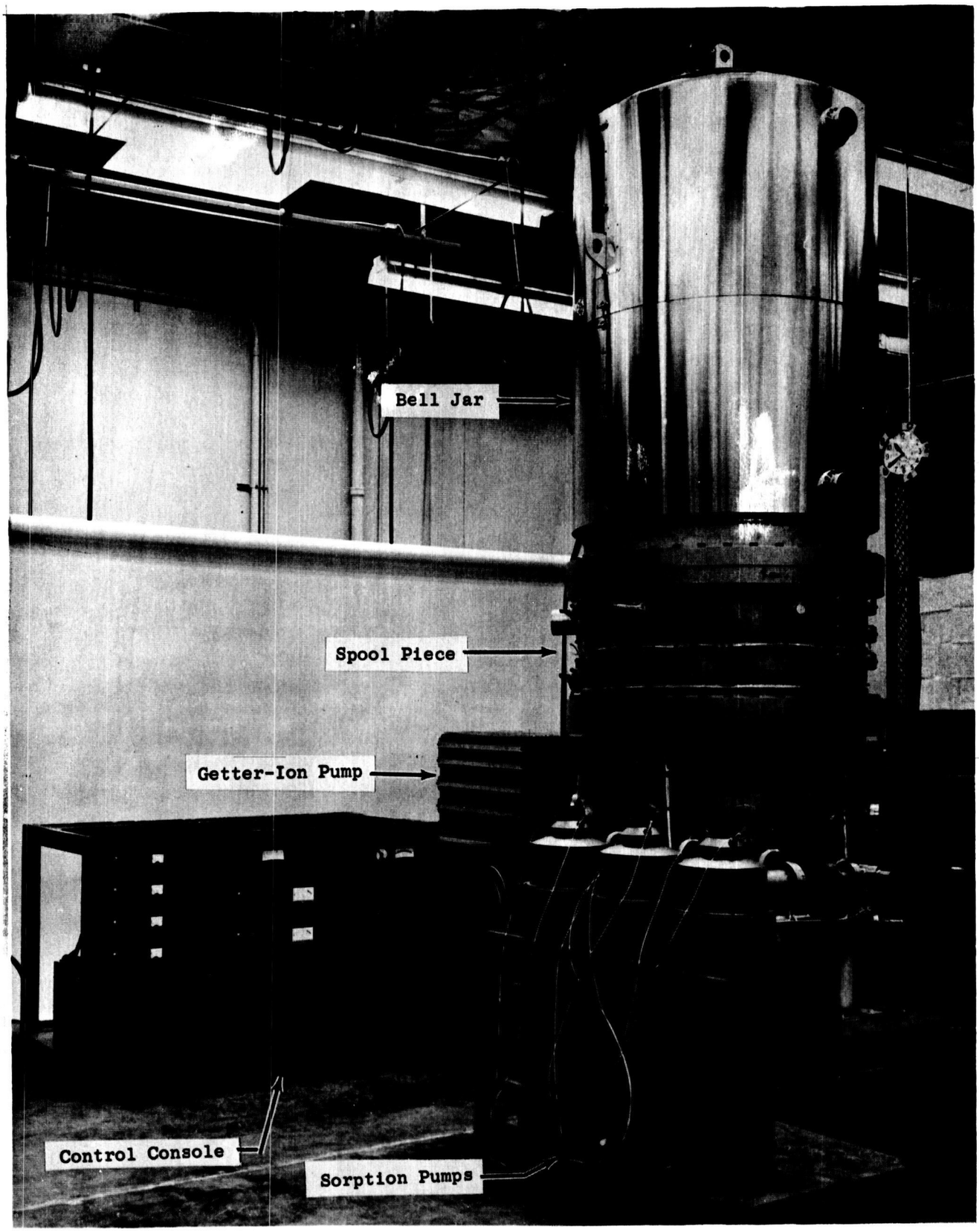

Figure 19. High Vacuum System (10^{-10} Torr Range) for Pre-prototype Loop. The Chamber is 48 Inches in Diameter and 128 Inches High and Incorporates a 2400 ℓ/sec Getter-Ion Pump, 20,000 ℓ/sec (H_2) Titanium Sublimation Pump and Liquid Nitrogen Sorption Pumps.

would rise from the 10^{-6} torr range to 15 microns in less than 10 minutes. Extensive leak checks using both helium and halogen detection equipment failed to locate a leak, and it was assumed that the poor performance was the result of outgassing of some foreign material in the pump cell. The pump cell was removed from the system and a detailed visual inspection failed to disclose any foreign material in the pump cell. The isolated pump cell was again leak checked but a leak could not be located even though the isolated pump cell had a high leak-up rate. The entire pump unit was shipped back to the vendor for further tests and repairs.

9. Refluxing Potassium Compatibility Tests

Two reflux capsule tests are to be conducted to evaluate possible mass transfer reactions that may occur in potassium between the turbine material, Mo-TZM alloy and the containment material, Cb-1Zr alloy, at 2000°F.

The capsules have been made from 1-inch OD x 0.080-inch thick wall Cb-1Zr alloy seamless tubing and are approximately 10 inches long, Figure 20. The ID of the capsules was gun drilled and honed to a 32 rms finish. Five, 1-inch long Mo-TZM alloy inserts having a 0.080-inch thick wall are located in the condensing zone of the capsules. The inserts were accurately machined to fit the ID of the capsule and also have a 32 rms finish. Thermocouple wells are located in the top and bottom of the capsules such that temperature measurements can be made in the center of both the boiling zone and the condensing zone.

Prior to testing, the Mo-TZM alloy inserts will be accurately weighed and measured; the ID of the capsules will be accurately measured; microstructure, microhardness, strength properties and chemical analyses for O, H, N and C will be determined and documented for both the Mo-TZM alloy and the Cb-1Zr alloy. The capsules will be filled with high-purity potassium under vacuum to a height of 3.75 inches. Subsequently, the tops of the capsules will be sealed immediately in the vacuum by electron beam welding techniques. The potassium used will be purified by slagging, filtering, vacuum distillation and hot trapping in a titanium lined, zirconium gettered hot trap. Samples of the potassium will be analyzed for oxygen by the mercury amalgamation method and for metallics by spectrographic techniques before and after purification and after the filling of the capsules. The latter samples will be obtained during the filling operation. After filling, the capsules will receive a careful visual and radiographic examination to assure their integrity.

The capsules will be tested in a high vacuum of 10^{-8} torr or better for 1,000 hours at a liquid temperature of 2000°F. The test setup is shown in the drawing in Figure 21. A split tantalum strip heater will be used to heat the liquid region of the capsule.

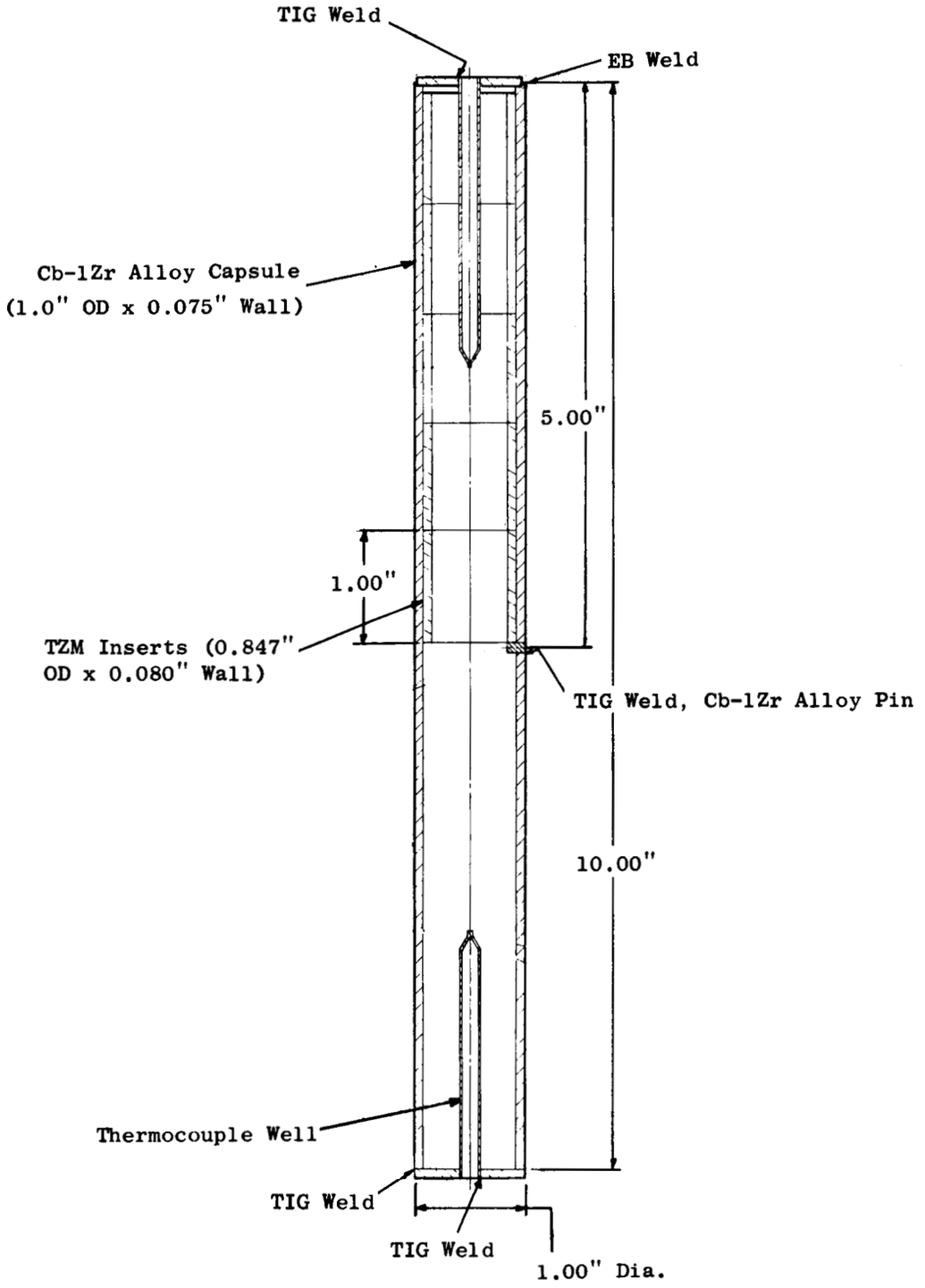

Figure 20. Cb-1Zr Refluxing Potassium Corrosion Capsule Containing TZM Alloy Inserts in Condensing Region.

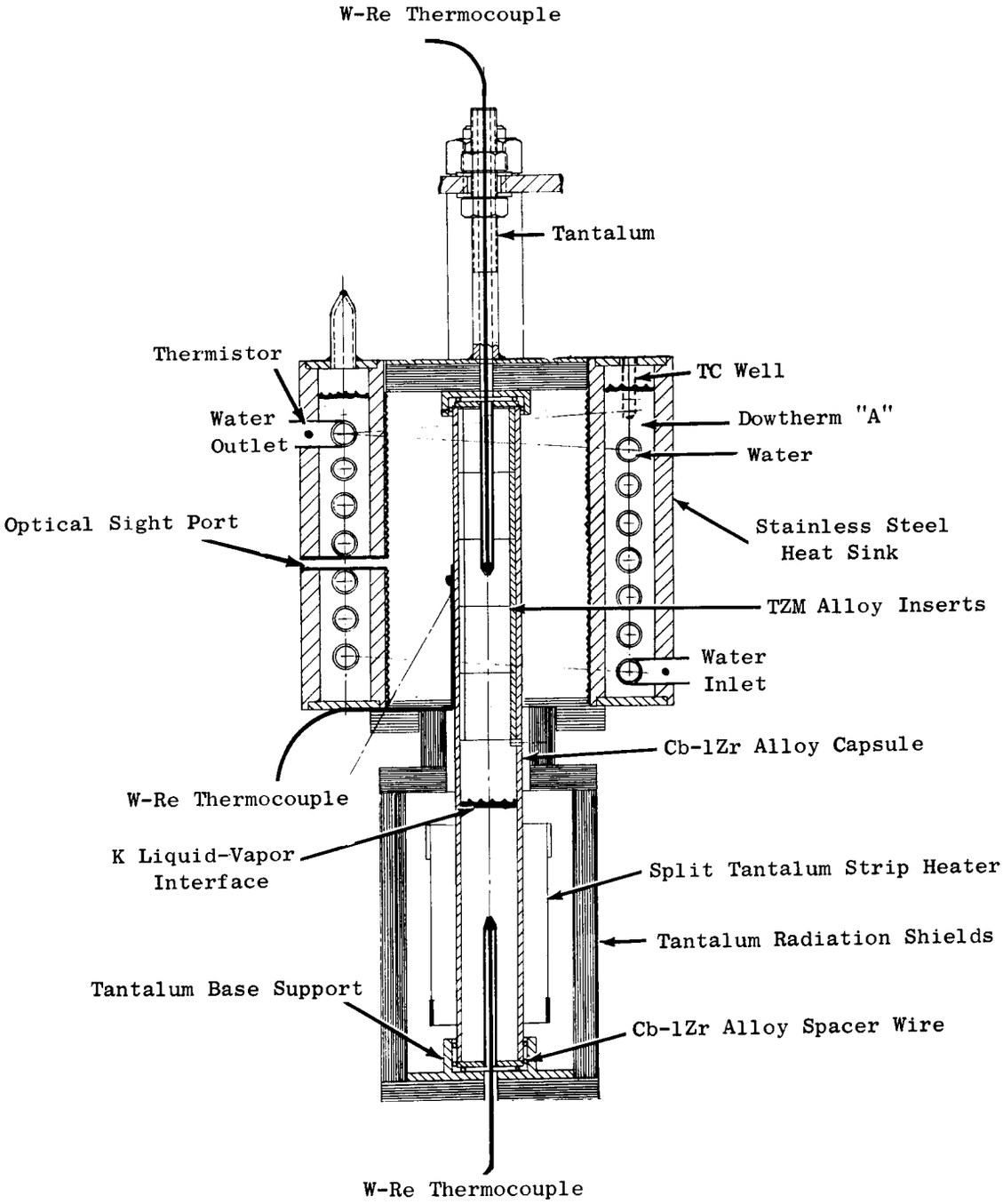

Figure 21. Test Arrangement for Reflux Capsule Testing in High Vacuum.

The condensing rate will be determined by two means. The heat rejected to the Dowtherm "A" filled, water cooled heat sink will be measured, and the condensing rate determined by dividing this value by the heat of condensation for potassium at the condensing temperature. Temperature of the water entering and leaving the Dowtherm jacket will be measured with thermistors inserted in the inlet and exit water lines. The temperature of the potassium will be measured by W+3%Re vs W+26%Re thermocouples located in the thermocouple wells. The rate will also be determined by means of a radiation calculation. The radiating surface temperature will be measured by using an optical brightness pyrometer and a W+3%Re vs W+26%Re thermocouple. All materials are on hand for the construction of the test facility.

10. Material Procurement

All of the Cb-1Zr alloy mill products ordered in the fall of 1963 for the construction of Loops I and II and the Pre-prototype Loop have been received and logged into the inventory. A list of these materials is given in Table IX. Inventory, quality assurance, and disposition information on all materials has been carefully documented. Inspection of the later shipments is continuing and, generally, most of the products have satisfied the material specification requirements of the program. In addition to the material listed in Table IX, orders have been placed for small quantities of Cb-1Zr alloy 0.062 and 0.094-inch diameter welding wire and for 0.040-inch thick sheet for weld test coupons. The latter specimens are required to fulfill the quality assurance provisions in all Cb-1Zr alloy welding operations. Also, small quantities of Mo-TZM alloy (0.125-inch diameter, 0.750-inch diameter and 1.250-inch diameter bar) and T-111 tantalum alloy (1.0-inch and 0.187-inch diameter bar and 0.015 and 0.020-inch thick sheet) were procured. The Mo-TZM material was produced to specification SPPS-15 (modified) by American Metal Climax Company for application in valves; the T-111 bar and foil was produced by National Research Corporation to preliminary specifications evolved from a combination of available information on the alloy and material application requirements and is to be used in the fabrication of high response pressure transducers for Loop II.

11. Grain Growth Studies on Cb-1Zr

A. Cb-1Zr Alloy Tube

Limited investigations were conducted to determine the effect of varying amounts of deformation and subsequent heat treatments on the grain coarsening of the 0.375-inch OD x 0.065-inch thick wall Cb-1Zr alloy tubing used in the fabrication of the Loops I and II.

The tubing utilized in the fabrication of Loop I was produced from a Cb-1Zr alloy tube hollow, 2-inch OD x 0.250-inch thick wall x 48-inch long, procured from the DuPont Metals Center. The hollow was produced from heat number 11-229-01 having the following analysis:

TABLE IX

SUMMARY OF Cb-1Zr ALLOY MILL PRODUCTS PURCHASED

Vendor	Mill Product, Size & Weight
Kawecki Chemical Company	Foil: 0.002" x 0.5" x 10,800' (40 lbs) 0.002" x 3.5" x 125' (3.2 lbs) 0.005" x 3.0" x 8' (0.44 lbs)
Stellite Division, UCC	Bar: 0.125" dia. (0.2 lbs) 0.375" dia. (1.6 lbs) 0.500" dia. (25 lbs) 1.00" dia. (29 lbs) 1.25" dia. (16 lbs) 1.50" dia. (26 lbs) 2.00" dia. (58 lbs) 0.5" x 1.0" (21 lbs) 1.0" x 1.0" (10 lbs)
Stauffer Metals Division	Bar: 2.50" dia. (69 lbs) 3.00" dia. (75 lbs) 3.75" dia. (67 lbs) 3.50" dia. (81 lbs)
Wah Chang Corporation	Sheet: 0.017" x 12" (5 lbs) 0.030" x 12" (8 lbs) 0.125" x 10" (62 lbs) 0.125" x 30" (84 lbs) Plate: 0.250" x 12" (44 lbs) 0.500" x 6" (104 lbs) Tube: 0.1875" OD x 0.025" wall (0.9 lbs) 0.250" OD x 0.062" wall (0.8 lbs) 0.375" OD x 0.065" wall (48 lbs) 1.0" OD x 0.100" wall (35 lbs) 2.75" OD x 0.125" wall (15.3 lbs) 3.25" OD x 0.125" wall (13.7 lbs)
Superior Tube	Tube: 0.375" OD x 0.008" wall x 7" long (200 pcs.)

Element, %				
Zr	C	N	O	H
1.0	0.0046	0.0015	0.0123	0.0006

The tube hollows were tube-reduced at room temperature to 0.875-inch OD x 0.095-inch thick wall, representing a reduction of 83%. Subsequently, the rocked tube, approximately 20 feet long, was pickled in a solution of 60%H_2O-20%HNO_3-20%HF, wrapped in tantalum foil and heat treated for 1 hour at 2200°F in a vacuum of 1 x 10^{-5} torr. After annealing, the tubing was reduced 78% by drawing at room temperature to the desired 0.375-inch OD x 0.065-inch thick wall x approximately 75 feet (8 feet maximum lengths). This material was given the same pickling and annealing treatments described above. Chemical analyses of a sample of the pickled and annealed tubing indicate the following levels of interstitial elements:

Element, %			
C	N	O	H
0.0020	0.0009	0.0136	0.0004

Microexamination of samples of the annealed tubing revealed a fine grain structure represented by an ASTM grain size of 6-8.

Twelve-inch lengths of the tubing were formed manually over 2.6-inch diameter and 3.5-inch diameter dies. These configurations approximate the degree of deformation the tubing would experience in the fabrication of Loop I. The maximum strain for both bends is shown in Figure 22. Two transverse sections were carefully cut from the center of the bend of the formed tubing so as to obtain as uniform an amount of strain from sample to sample within a single bend as possible. After a pickling treatment, one sample of each bend diameter was wrapped in tantalum foil, placed in a tantalum container and exposed for 165 hours at 2200°F in a vacuum of 1 x 10^{-5} torr. A second set of specimens was exposed to 2000°F for the same length of time and similar environmental conditions.

Subsequent metallographic examination of the heat treated samples revealed significant preferential grain growth over a considerable area on both the tension and compression side of the bends. Figure 23 shows a macrograph and a 100X micrograph of the sample formed over the 3.5-inch diameter die and heat treated for 165 hours at 2200°F. There was little difference in the magnitude of the preferential grain growth between the two bend diameters at a given temperature. However, the total volume of grain growth was less in those samples exposed at the lower temperature. The fine-grained structure shown in Figure 23 is representative of the structure of the recrystallized tubing.

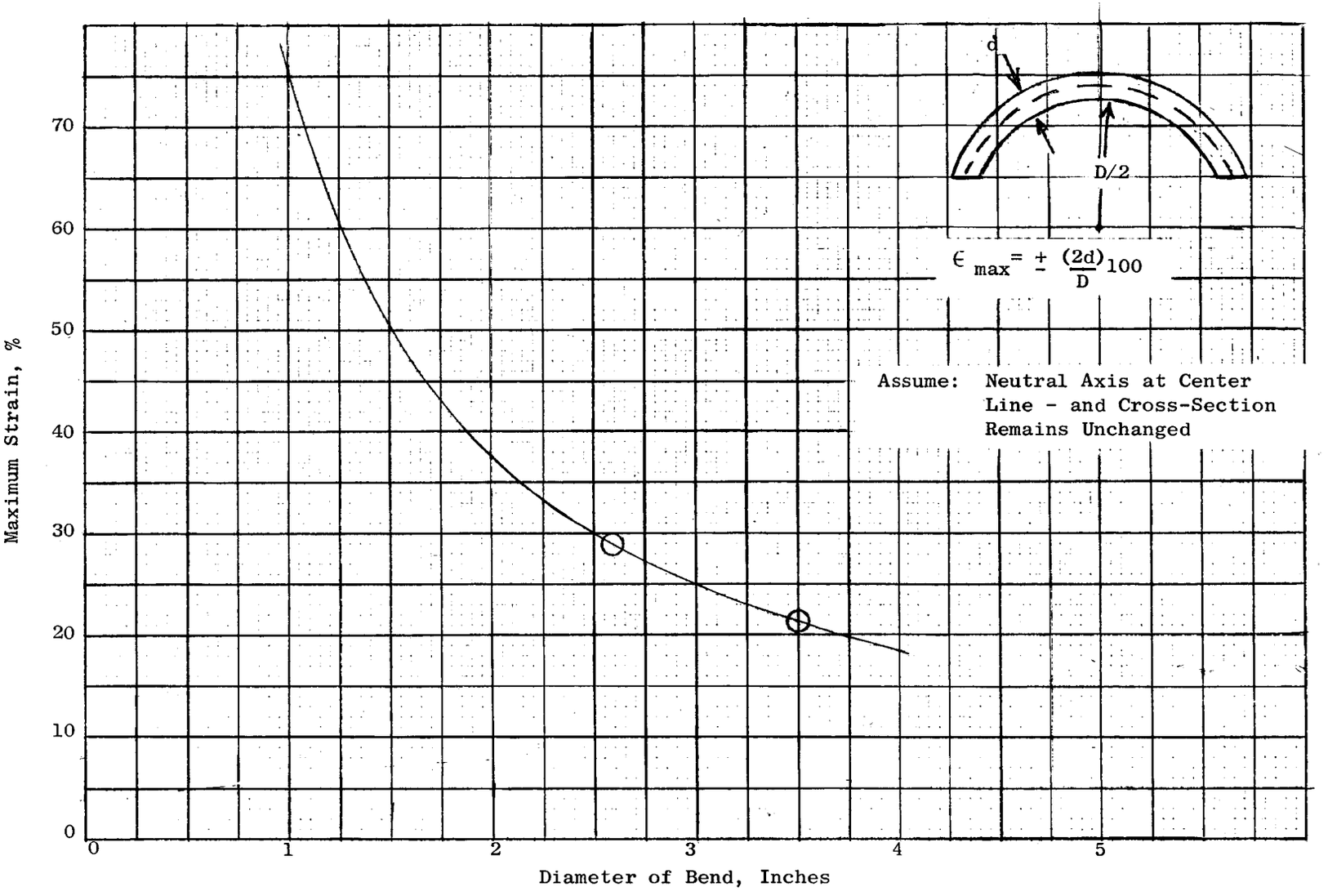

Figure 22: Effect of Bend Diameter on the Maximum Fiber Strain in 0.375-Inch OD x 0.065-Inch Thick Wall Tubing.

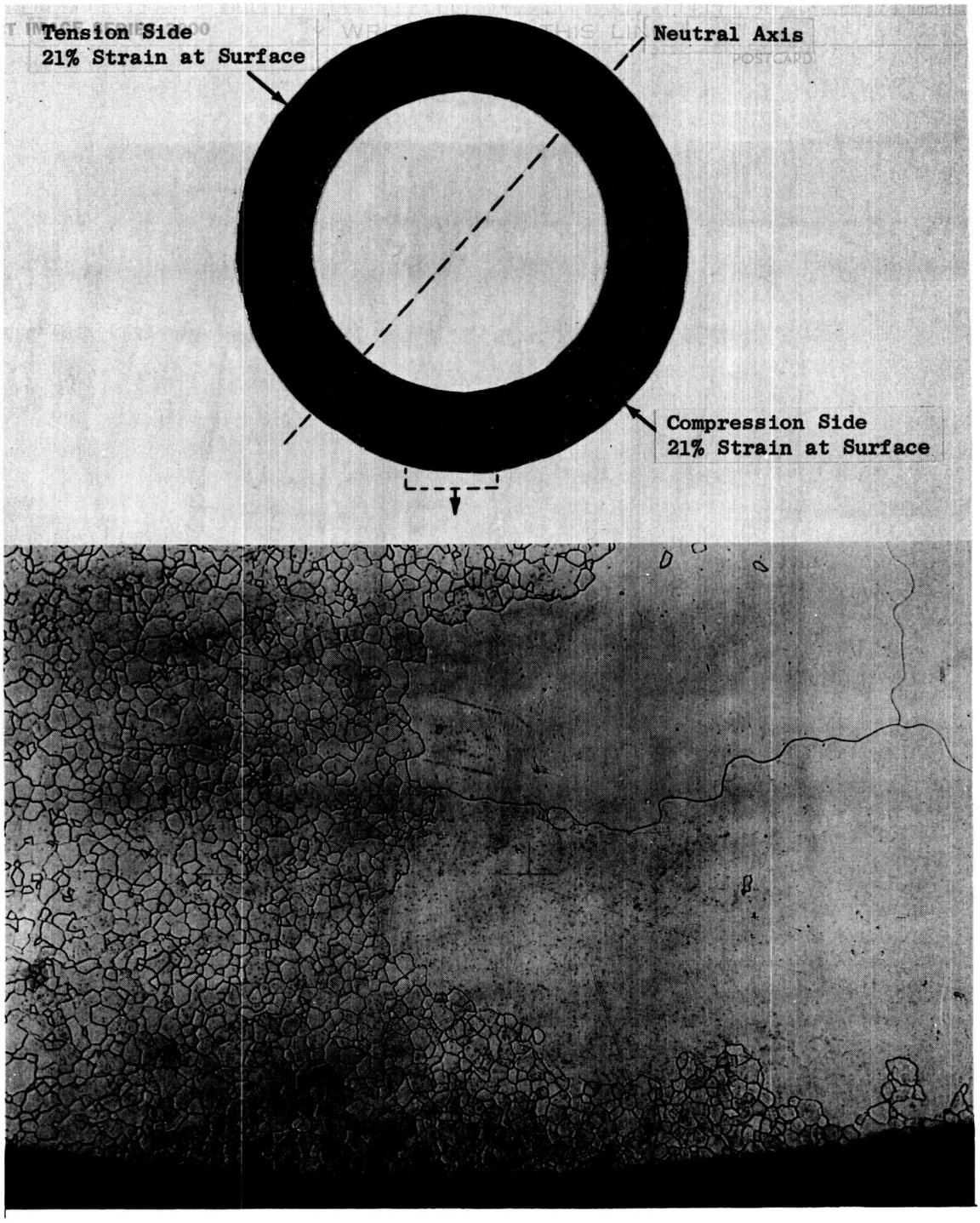

Figure 23. Macrograph and Micrograph of a Cross Section of 0.375-Inch Diameter x 0.065-Inch Thick Wall Cb-1Zr Alloy Tubing. The Recrystallized Tubing was Bent Around a 3.5-Inch Diameter Die and Subsequently Annealed for 165 Hours at 2200°F.

(Top - C64021002, Bottom - K352)

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 8X Top
100X Bottom

In order to determine whether this condition exists after a short time at elevated temperatures, one section of each bend diameter was vacuum heat treated for 1 hour at 2200°F and examined for evidence of grain growth. Initiation of growth was found only on the tension side of the sample formed over the 2.6-inch diameter die, as illustrated in Figure 24.

Because of the disconcerting results of this limited investigation, it was decided to perform similar experiments with the major lot of 0.375-inch OD tubing produced by the Wah Chang Corporation to specification SPPS-2B for Loop II and the Pre-prototype Loop.

The routine check analysis of the as-received tubing from Wah Chang indicated the following levels of interstitial elements:

Heat No. 98-70546

Element, %			
C	N	O	H
0.0055	0.0059	0.0142	0.0006

Fortunately, several lengths of tubing were in the as-drawn condition which afforded the opportunity of investigating the effects of several prior heat treatments as well as post-forming anneals on the grain growth behavior of the material. Two bend diameters, 2.6 and 10 inches, were chosen for this study.

Two six-inch lengths and two twelve-inch lengths were cut from one of the as-received tubes. These blanks were pickled, as previously described, and one section of each length was wrapped in tantalum foil and heat treated for 1 hour at 1800°F in a vacuum of 1×10^{-5} torr. The remaining blanks were vacuum annealed for 1 hour at 2200°F. After the lengths of tubing were heat treated, the shorter blanks were formed over the 2.6-inch diameter die and the twelve-inch pieces over the 10-inch diameter die. Subsequently, transverse samples were sectioned from the center of the bend, pickled and wrapped in tantalum foil. One set of samples, consisting of a specimen from each bend and prior heat treatment, was exposed for 1 hour at 2200°F and another set for 100 hours at 2200°F, both in vacuums of 1×10^{-5} torr.

Microexamination of surfaces of specimens sectioned from the samples annealed for 1 hour at 2200°F revealed no significant change of the grain size as a result of the deformation and annealing. Although the samples that were exposed for 100 hours showed no evidence of the gross amount of preferential grain growth that was found in the earlier lot of tubing, a small amount of grain growth was observed on those samples given the 1 hour at 1800°F vacuum stress-relief and then formed over either the 2.6-inch or 10-inch diameter die (Figure 25). In this case, the grain growth observed was limited to a uniform 2 to 5 mil layer that extends

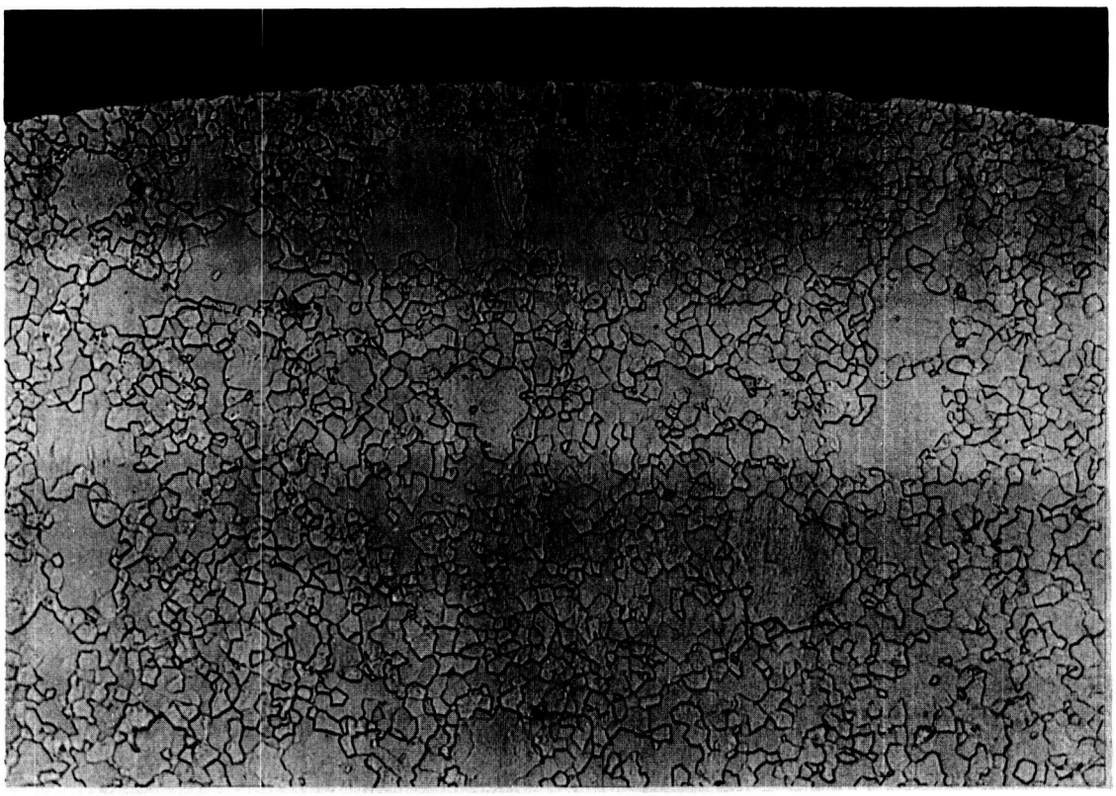

Figure 24. Micrograph of a Cross Section of 0.375-Inch Diameter x 0.065-Inch Thick Wall Cb-1Zr Alloy Tubing. The Recrystallized Tubing was Bent Around a 2.6-Inch Diameter Die and Subsequently Annealed for 1 Hour at 2200°F.

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 100X (K1210)

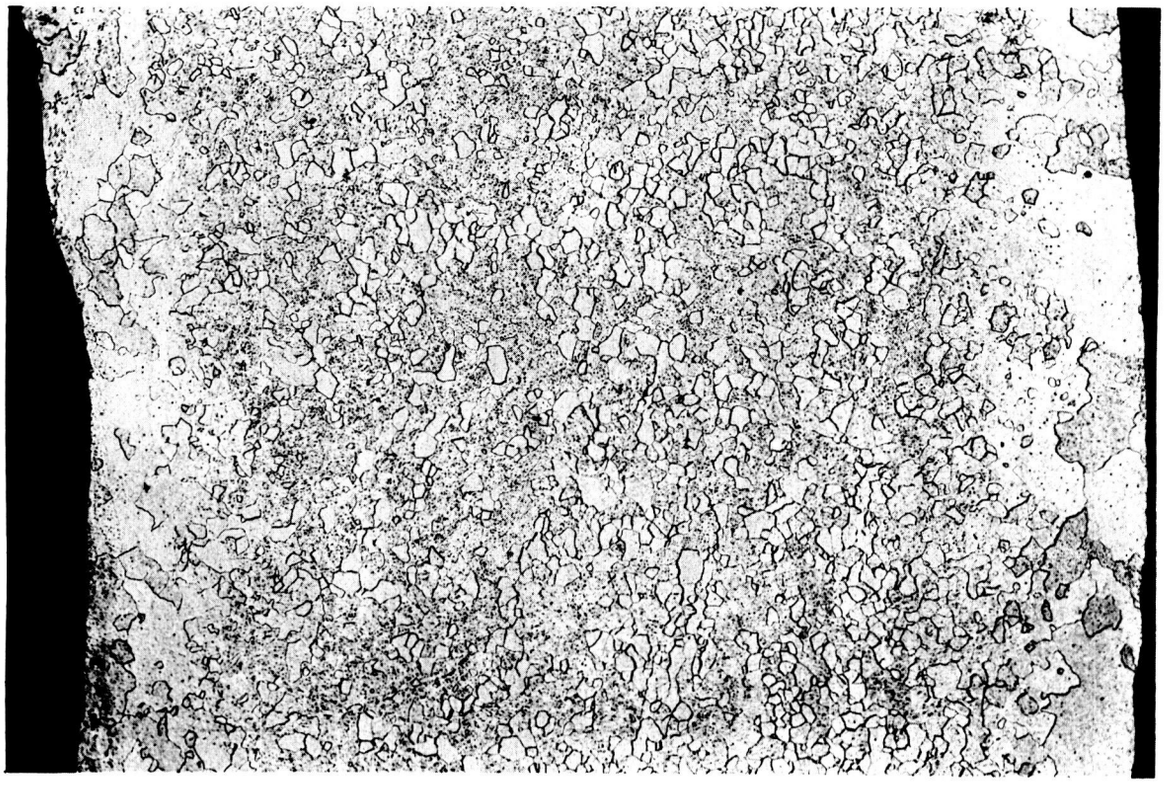

Figure 25. Micrograph of a Cross Section of a 0.375-Inch Diameter x 0.065-Inch Thick Wall Cb-1Zr Alloy Tubing. The As-Drawn Tubing was Heat Treated for 1 Hour at 1800°F in a Vacuum of 1×10^{-5} Torr, Formed Over a 2.6-Inch Diameter Die and Subsequently Annealed for 100 Hours at 2200°F in a Vacuum of 1×10^{-5} Torr. Note Grain Growth Near the ID (Left) and OD (Right) of the tube wall.

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 100X (K2226)

completely around the outer and inner periphery of the cross section of the tube. This effect was not observed in any of the specimens that were given a prior 2200°F anneal. From this limited investigation a decision was made to vacuum heat treat all of the as-drawn tubing for 1 hour at 2200°F prior to fabrication into components for Loop II and the Pre-prototype Loop.

In reviewing the histories of the two lots of tubing, two major differences become apparent:

1. The earlier lot of tubing received considerably more cold work (78%) between the last in-process anneal and the final anneal than the latter lot (20-40%).

2. A lower level of total interstitial elements was found in the tubing exhibiting the preferential grain growth:

	Element, ppm				
	C	N	O	H	Total
DuPont Heat No. 11-229-01 (First Lot)	20	9	136	4	169
Wah Chang Heat No. 98-70546 (Second Lot)	55	59	142	6	262

Since the material from the first lot of tubing was completely recrystallized before being formed, the most probable reason for the different behavior is the difference in the interstitial element concentrations, especially carbon and nitrogen. Use of the first lot of tubing in the fabrication of Loop II will be restricted to non-critical components operating at low temperatures, and none of this material will be used in the construction of the Pre-prototype Loop.

Additional studies have been initiated to establish the magnitude of strain necessary to induce grain coarsening or abnormal growth in the Cb-1Zr alloy. Samples of Cb-1Zr alloy bar, 1-inch thick x 0.5-inch wide x 4-inch long, were machined into wedge-shaped specimens tapering from 0.750-inch to 0.062-inch thick. These wedges were press forged at room temperature to approximately 0.180-inch thick resulting in a deformation gradient in the material of 0 to 76%. The forged specimens will be sectioned lengthwise, heat treated at various time/temperature combinations and metallographically examined to locate any area of critical strain that results in abnormal grain growth. To-date the first 100-hour exposure has been completed and metallographic preparation is underway.

B. Cb-1Zr Valve Bellows and Pressure Transducer Diaphragms

A brief investigation was undertaken to evaluate the possiblity of grain coarsening in the 0.005-inch thick transducer diaphragms and in 0.008-inch thick bellows for the valves since these parts are subjected to varying amounts of strain and short time/high temperature thermal exposure during the various phases of fabrication. A sample diaphragm had been electron beam welded into a transducer housing to establish proper welding parameters. This component was then post-weld heat treated for 1 hour at 2200°F in a vacuum of 1×10^{-5} torr and sectioned for metallographic evaluation. Examination of a section containing a typical diaphragm convolution indicated a fine grain structure with no indications of coarsening. Figure 26 shows the typical microstructure of the formed and heat treated diaphragm, compared to the as-formed diaphragm.

A completely formed bellows was sectioned lengthwise and one half was given a 2-hour treatment at 2200°F in a vacuum of 1×10^{-5} torr. The as-formed and the heat-treated structures of the outer convolution region of the bellows are shown in Figure 27. No significant change in grain size occurred as a result of the heat treatment.

Microhardness measurements were made on longitudinal and transverse sections of the bellows after each stage of fabrication and heat treatment. The Knoop hardness (100-gram load) varied from 100 to 145 following the final forming operation and from 80 to 110 following heat treatment for 2 hours at 2200°F.

The obvious difference in the microstructure between the transducer diaphragm and the bellows after post-forming heat treatments is believed to be the result of considerably higher interstitial element concentrations in the bellows material which effectively raises the recrystallization temperature. The as-received analysis of these materials is given in Table X.

12. Helium Analysis System

The helium analysis system described in the last quarterly report[2] has been moved to a position adjacent to the welding chamber and sample lines have been installed. The mercury diffusion pump is now being cooled by a recirculating, refrigerated bath so that, except for electrical power, the system is independent of utilities.

Residual gas total pressure in the system has been reduced to about 3×10^{-10} torr, as indicated on the ion gauge. About 90% of this residual gas is hydrogen. This reduction was accomplished by additional

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2 Potassium Corrosion Test Loop Development, Quarterly Progress Report 2, Covering the Period October 15, 1963 through January 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54008.

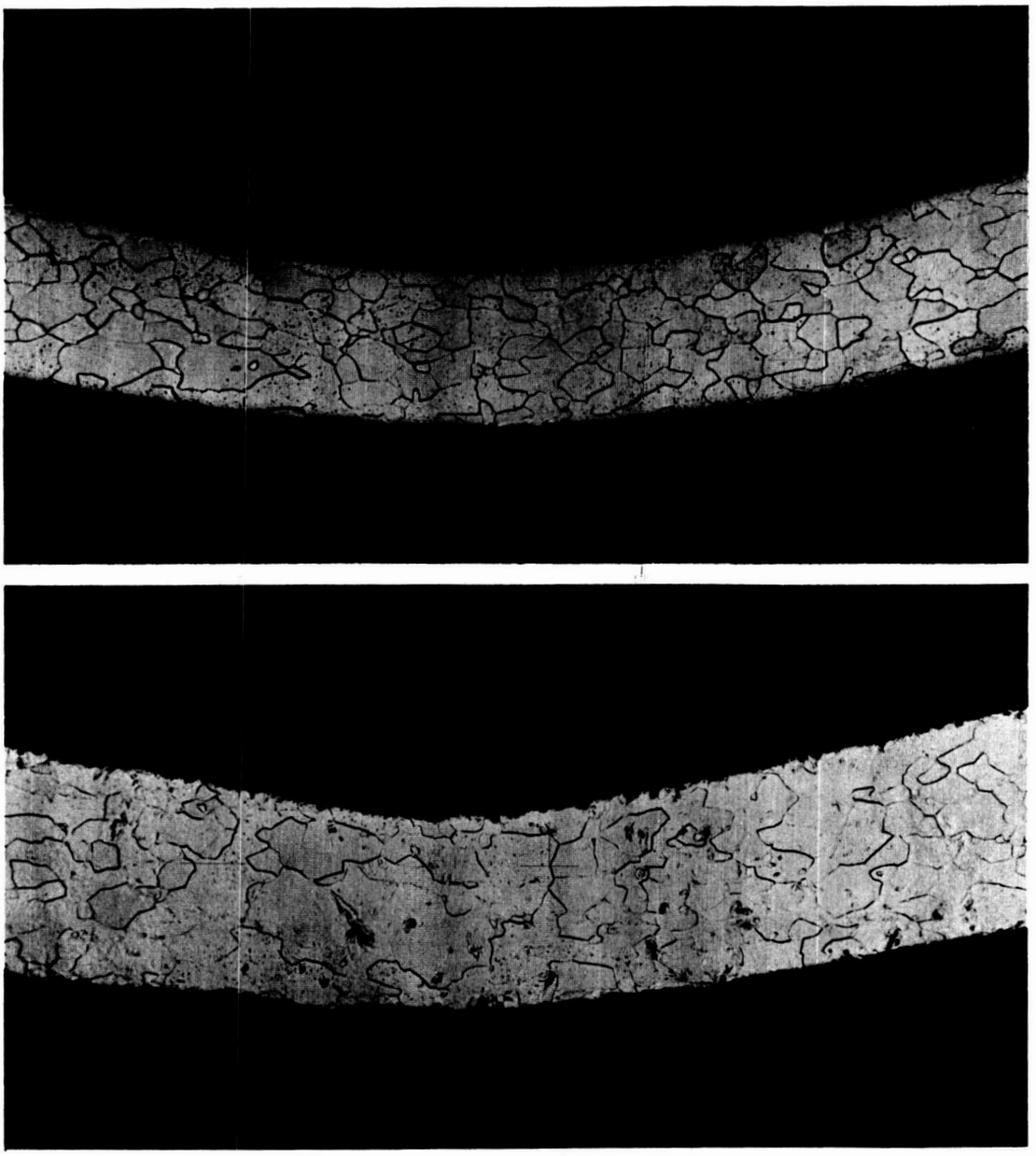

Figure 26. Microstructure of Formed 0.005-Inch Thick Cb-1Zr Alloy Diaphragm for Taylor Pressure Transducer. (Top) As-Formed: (Bottom) After a 1-Hour Anneal at 2200°F in a Vacuum of 1×10^{-5} Torr.

(Top - K2223, Bottom - K1954)

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 250X

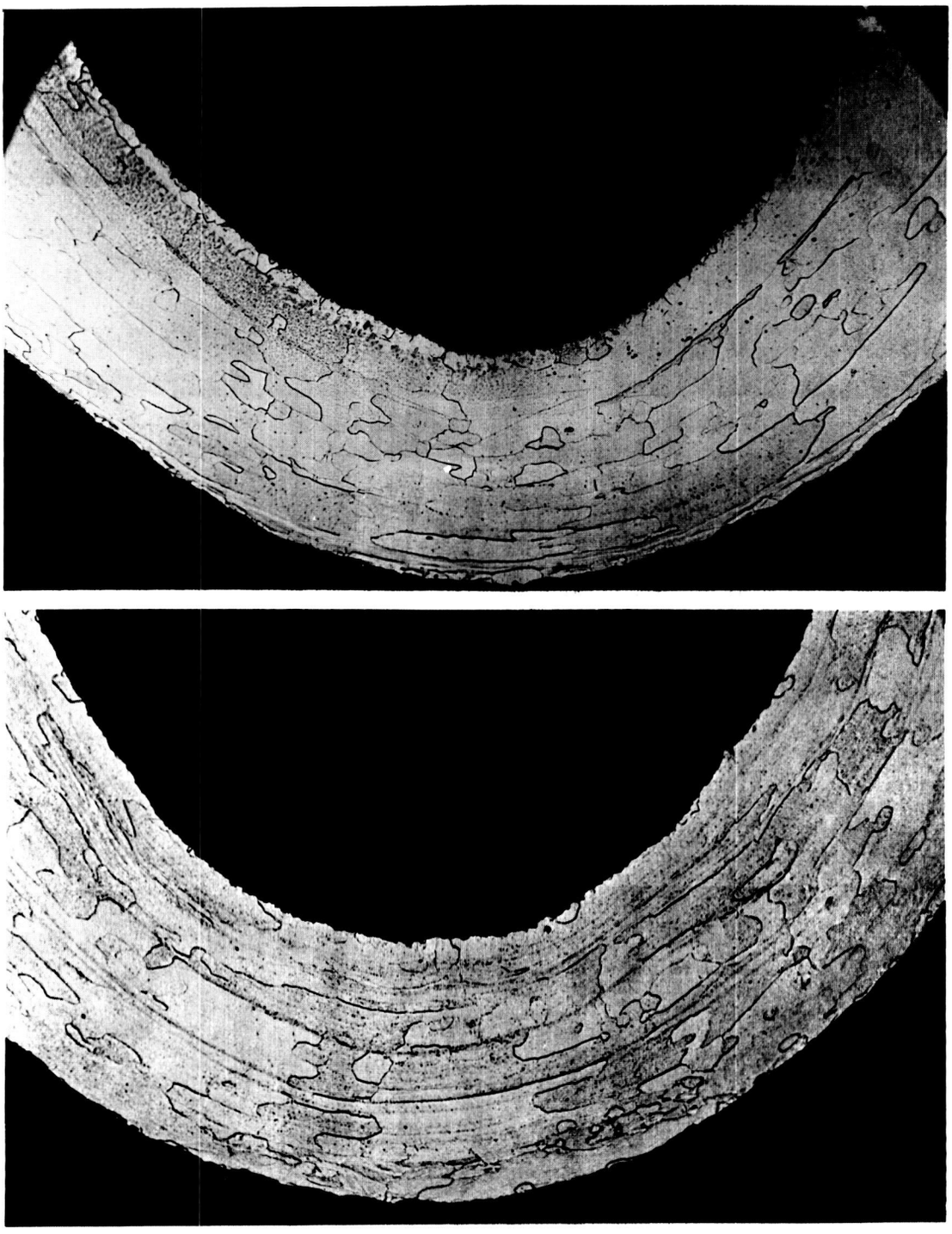

Figure 27. Microstructure of Fully Formed Cb-1Zr Alloy Bellows. Top, As-Formed: Bottom After a 2-Hour Anneal at 2200°F in a Vacuum of 1 x 10^{-5} Torr.

(Top - K222, Bottom - K1080)

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 250X

TABLE X

AS-RECEIVED ANALYSES OF FOIL FOR PRESSURE TRANSDUCER DIAPHRAGM AND TUBING FOR VALVE BELLOWS FABRICATION

	Vendor	Condition	Heat No.	Element, % C	O	N	H	Total
Cb-1Zr Alloy 0.005" Foil for Transducer	Kawecki	2200°F/1 hr	333-5	0.010	0.0115	0.0056	0.0001	0.0272
Cb-1Zr Alloy 0.375" OD x 0.008" Wall Tube for Bellows	Superior Tube	2300°F/1 hr	3.55-70274	0.015	0.0827	0.0106	0.0002	0.1085

bakeout of the system, location and repair of a small leak in the foreline, and by continued pumping on the system.

The partial pressure analyzer was calibrated for H_2, He, N_2 and A by comparison with the ion gauge. The sensitivity of the analyzer was found to vary markedly with mass. The senitivity to other gases is obtained from the four calibration points.

The system and vacuum achieved is now sufficient to detect impurities in helium at levels below 1 ppm with the exception of hydrogen. The detection limit for H_2 is estimated to be about 10 ppm at the present time. Analyses of helium from the welding chamber are now being obtained on a routine basis. The accuracy of the results, however, has not been determined. Present plans are to obtain a helium sample of certified impurity content with which to compare the results obtained with the partial pressure analyzer.

A. Calibration of the Partial Pressure Analyzer

A tentative calibration of the partial pressure analyzer has been obtained by injecting pure gases into the system and comparing the positive ion current from the spectrometer with that from the ion gauge. For the hydrogen calibration, the H_2 pressure in the system was increased by closing the foreline valve and allowing the pressure to build up. By this method the H_2 pressure could be changed by a factor of 40 without appreciable change in the other residual gases in the system. For the other calibration gases A, He and N_2, data have been obtained over a pressure range of at least two decades.

An important consideration in this type of calibration is the linearity of both the ion gauge and mass spectrometer ion current with pressure. Recent measurements by Davis[3] have shown the linearity of the Bayard-Alpert type gauge for He, A and N_2 from about 10^{-3} torr to near the X-ray limit of the gauge (10^{-10} torr). In this same study a mass spectrometer similar to the partial pressure analyzer being used on the welding chamber was found to have an output linear with pressure from the lowest pressures obtained (about 10^{-12} torr) well into the 10^{-5} torr region for helium. For nitrogen, linearity was maintained to about 10^{-7} torr.

The sensitivity of the ion gauge and the mass spectrometer for a particular gas is defined as

$$S = \frac{I}{i\ P} \qquad (1)$$

where I is the positive ion current resulting from a pressure P (torr) in the ionizing region

[3] W. D. Davis, "Gauge Calibration in the Ultra High Vacuum Range," GE Research Lab, Report No. 63-RL-35126, November 1963.

with an ionizing electron current, i. The factor to be obtained from the calibration is the ratio of the sensitivity of the mass spectrometer, S(ms), to the sensitivity of the ion gauge, S(ig). If the same ionizing electron current is used for both the ion gauge and the mass spectrometer, then the sensitivity ratio, R, is equal to the positive ion current ratio, thus:

$$R = \frac{S(ms)}{S(ig)} = \frac{I(ms)}{I(ig)} \qquad (2)$$

A typical plot of I(ms) vs I(ig) is shown in Figure 28. These data were obtained for argon at pressures between 10^{-9} and 10^{-7} torr. The sensitivity ratio is the slope of the line in Figure 28. Similar data have been obtained also for He, H_2 and N_2.

The sensitivity ratio for other gases may be obtained from these values if it is assumed that R is a function only of mass. Figure 29 is a plot of the sensitivity ratio (log scale) against mass number of the parent peak. The two curves of Figure 29 were obtained with electron multiplier voltages of 1275 and 1530 volts. The higher electron multiplier voltage results in an increase in gain by a factor of about 3. The very rapid change of R with mass is quite obvious from these curves. This mass dependent discrimination arises from the fact that masses are focused electrostatically and as a result, a relatively large number of ions are drawn from the source region at the very high potentials necessary to focus the low mass gases. While this is not a very serious difficulty in the use of the instrument once calibrated, it can be rather misleading if one assumes that relative ion currents approximate the relative concentrations.

The relative ion gauge sensitivity for various gases is defined as

$$r = \frac{\text{ion gauge sensitivity for gas}}{\text{ion gauge sensitivity for } N_2} \qquad (3)$$

The nominal sensitivity to nitrogen of the ion gauge used in the present study is 10 torr^{-1}. By combining equations (1), (2) and (3), the partial pressure of a particular gas may be obtained.

$$P = \frac{I(ms)}{10\ r\ i\ R} = I(ms)\ C \qquad (4)$$

where $C = \frac{1}{10\ r\ i\ R}$ is constant for each gas.

B. Residual Gases in the System

Typical mass spectra of the residual gases in the system are shown in Figures 30 and 31. In Figure 30 the most prominent peaks are at masses 16.3 and 19.3 and these, along with minor peaks at 23.3, 35.5, 37.5 and 39.5, probably do not arise from residual gases in the system. The origin

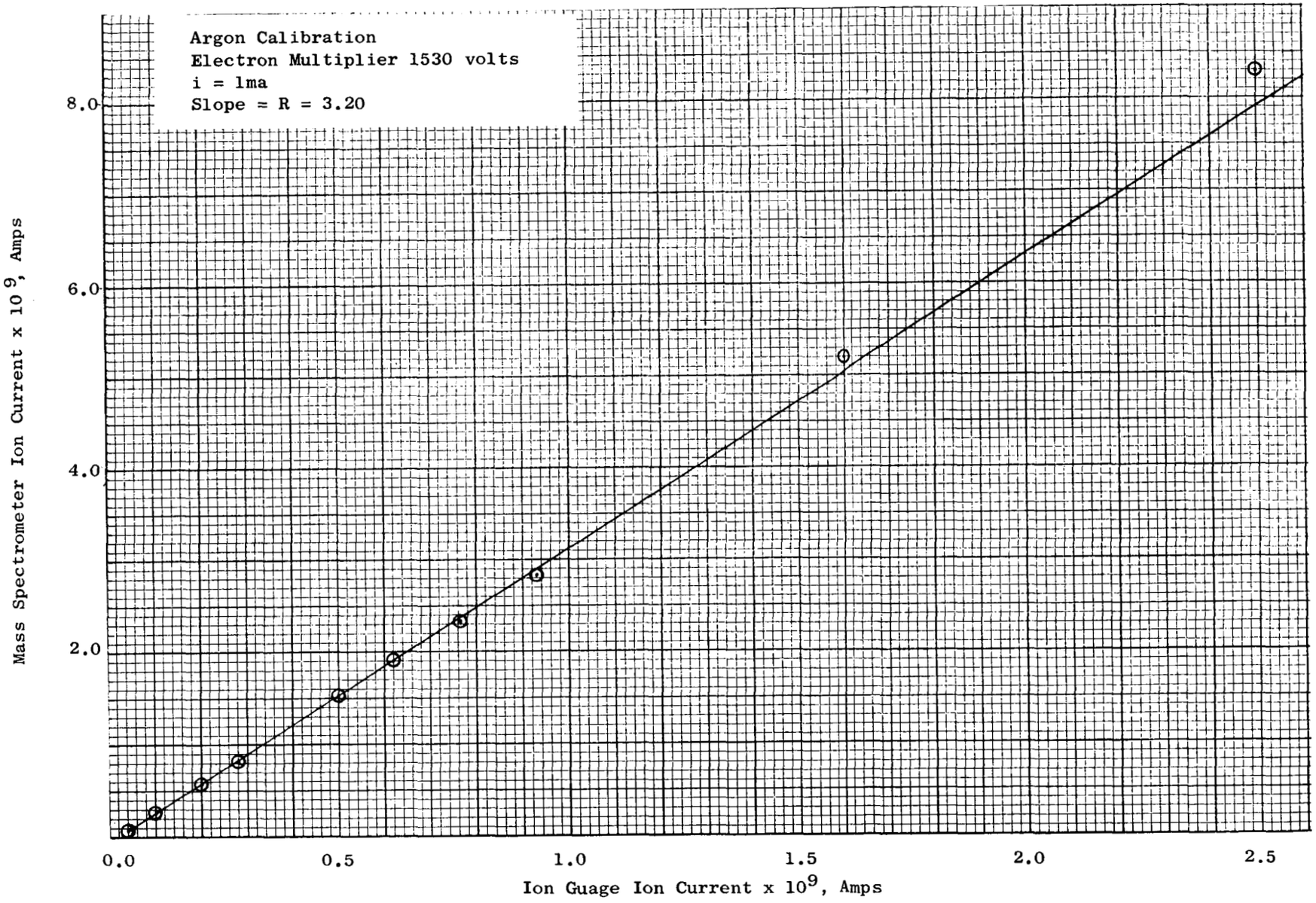

Figure 28. Mass Spectrometer Calibration for Argon.

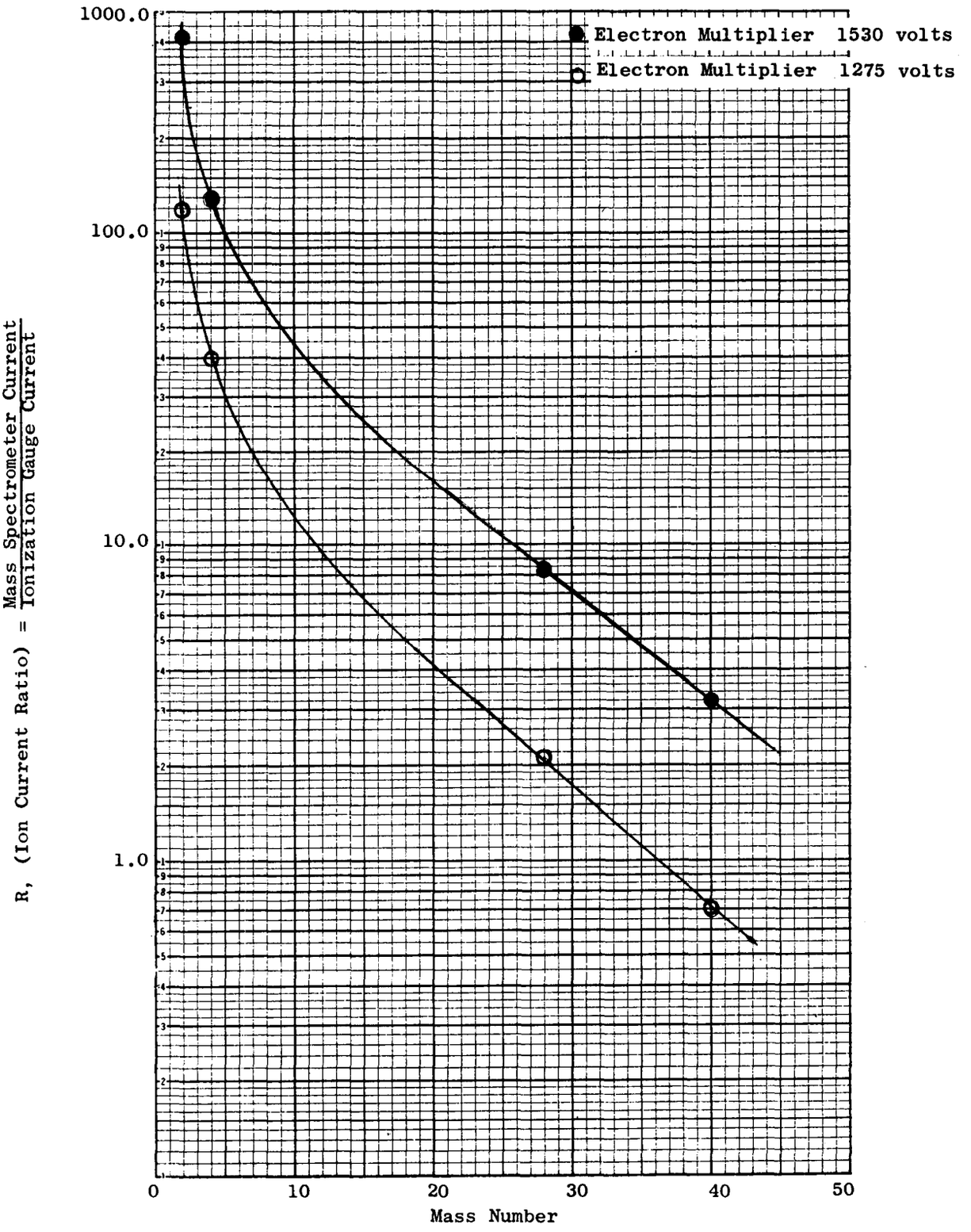

Figure 29. Ion Current Ratios Plotted Against Mass of Parent Peak for the Four Calibrating Gases.

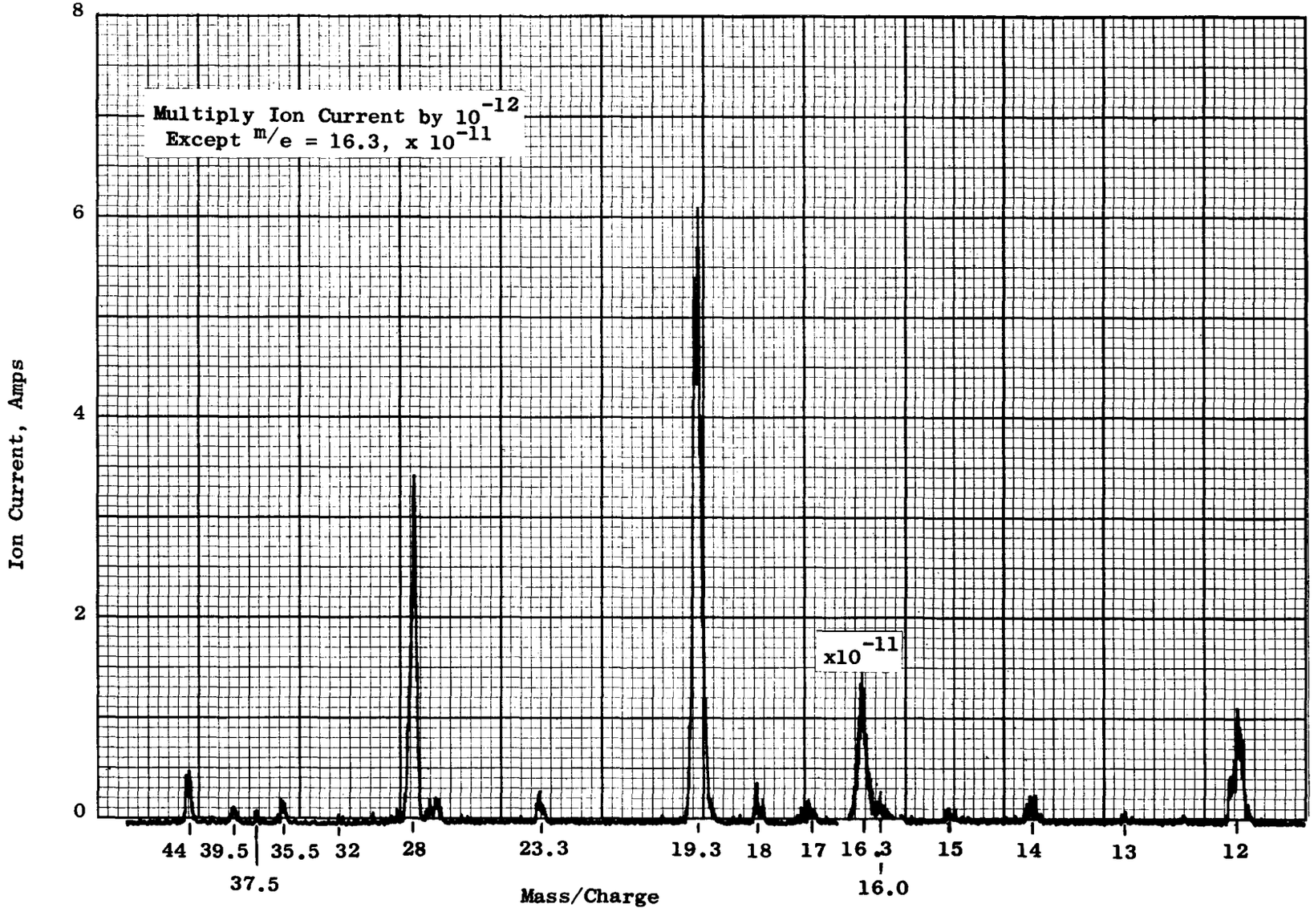

Figure 30. Residual Gas Mass Spectrum - High Mass Scan.

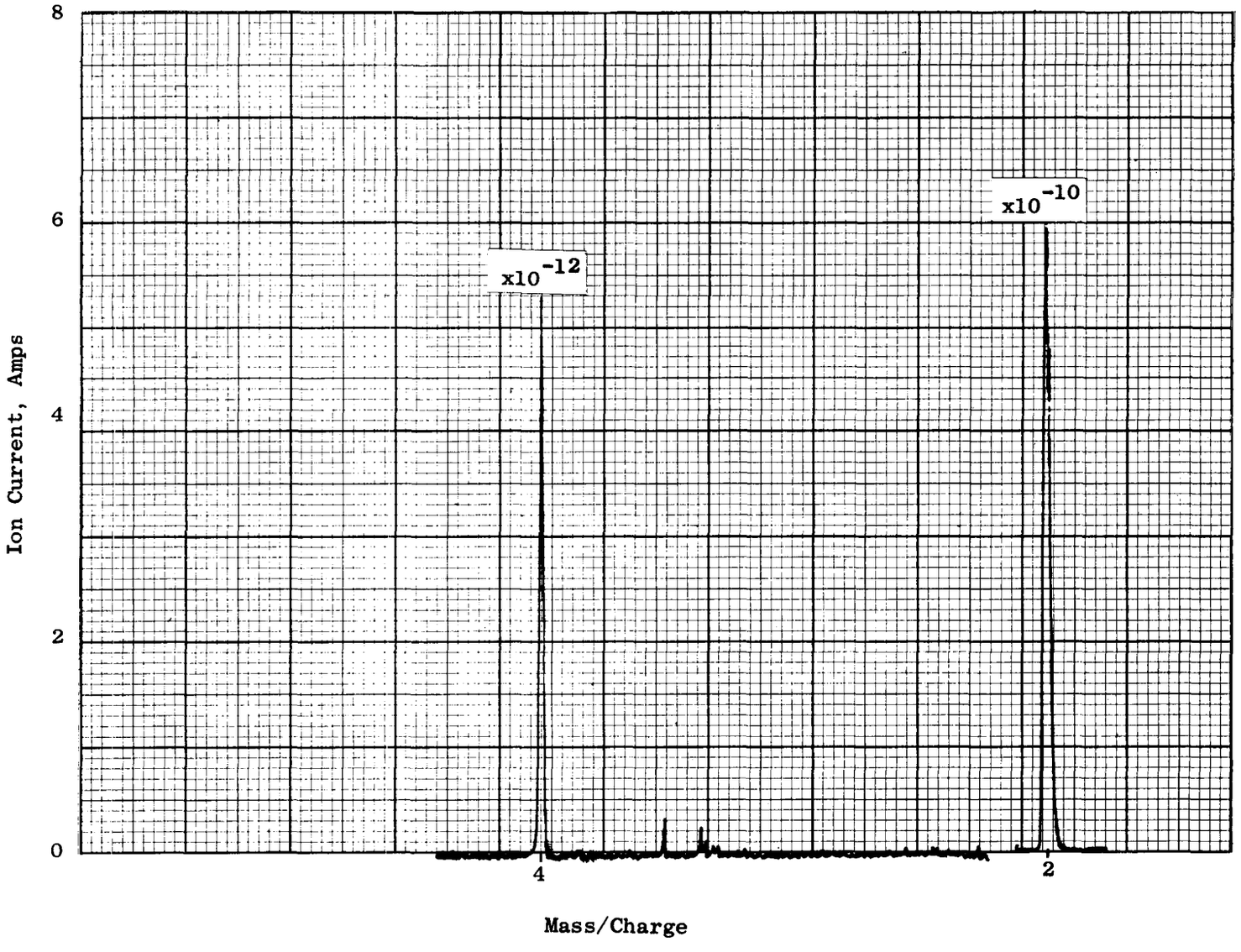

Figure 31. Residual Gas Mass Spectrum - Low Mass Scan.

of these peaks has been discussed by Davis[4] who concludes that the most probable source of these ions is by ionization of surface impurities in the source region by electrons striking the surface. This seems to be a quite reasonable explanation since the peaks do not occur at integral mass numbers, and, in addition, do not vary appreciably with total pressure in the system except for the 16.3 peak which is due to O^+ from absorbed CO^+. The other peaks are, according to Davis, due to Cl^+ (35.5 and 37.5), K^+ (39.5), Na^+ (23.3) and F^+ (19.3). The K^+ peaks can interfere with the determination of argon at very low concentrations. However, since argon is of no particular interest as an impurity in helium in the present study, this presents no problem. The large peak at 16.3 usually overlaps the 16 peak (O^+ and CH_4^+). This, too, presents no serious problem since reasonably accurate interpretation of the spectra can usually be obtained without the 16 peak.

Table XI shows the partial pressures calculated from the spectra of Figures 30 and 31. Values for R were obtained from the curve of Figure 29 for an electron multiplier voltage of 1530 volts. Values for r were obtained from various literature sources. Partial pressures were calculated from equation (4). The CH_4 ion current is estimated from the $\frac{m}{e} = 15$ peak and the N_2 ion current is estimated from the $\frac{m}{e} = 14$ peak. The sum of the partial pressures is 4.0×10^{-10} torr.

Partial ion gauge ion currents calculated from these data are shown in the last column of Table XI. The total calculated ion gauge current, 2.1×10^{-12} amp, may be compared with the measured value 4.2×10^{-12} amp. Thus, the measured ion gauge current is not completely accounted for by the mass spectra. There are several possible explanations for this apparent discrepancy. In the first place, spurious ions, similar to those observed in the mass spectra, may also contribute to the ion gauge current. Secondly, the ion gauge might have a tendency to read high at these low pressures which approach the X-ray limits of the gauge, and, finally, these could be gaseous impurities (such as mercury from the pump) that are not detected by the mass spectrometer.

C. Helium Analysis

In the analysis of helium, the residual gas mass spectrum is obtained and the residual gas ion currents are subtracted from the corresponding values obtained when helium is introduced into the system. It has been found that helium may be admitted to a total pressure of about 1×10^{-5} torr without excessive background currents due to scattered ions. For this total pressure a residual gas partial pressure of about 1×10^{-11} torr gives a mass spectrometer ion current equivalent to that from 1 ppm

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4 W. D. Davis, 1962 Transactions of the Ninth Vacuum Symposium, pp. 363, the MacMillam Company, New York (1962).

TABLE XI

TYPICAL RESIDUAL GAS PARTIAL PRESSURE CALCULATED

FROM THE MASS SPECTRA

	I(ms), Amp	R	r	C, $\frac{\text{Torr}}{\text{Amp}}$	P, Torr	Partial Ion Gauge Current Calculated Amp
H_2	5.9×10^{-10}	416	0.42	0.57	3.4×10^{-10}	1.4×10^{-12}
He	5.3×10^{-12}	127	0.19	6.6	3.5×10^{-12}	4.2×10^{-14}
CH_4	1×10^{-13}	23	1.07	4.1	4×10^{-13}	4×10^{-15}
H_2O	2×10^{-13}	19	0.89	6.0	1.2×10^{-12}	1×10^{-14}
CO	3.3×10^{-12}	8.3	1.07	11.3	3.7×10^{-11}	4×10^{-13}
N_2	3×10^{-13}	8.3	1.00	12.1	3.6×10^{-12}	4×10^{-14}
CO_2	4×10^{-13}	2.34	1.37	31.3	1.3×10^{-11}	1.7×10^{-13}
TOTAL					4.0×10^{-10}	2.1×10^{-12}

impurity in the helium. As can be seen in Table XI, it would be desirable to reduce the residual gas partial pressures due to CO, CO_2 and H_2, all of which are greater than 10^{-11} torr. Further reduction can probably be obtained by additional baking of the system, outgassing of the ion gauge and prolonged operation of the hot filaments.

The main question in the analysis of helium is whether or not the impurity concentration is maintained in the sampling and analysis procedure. Impurities such as O_2 and H_2O might be adsorbed in the walls of the system or, perhaps, react with the elements of the spectrometer. In order to determine the extent to which such effects may occur, a helium sample of known impurity content will be obtained with which results from the partial pressure analyzer may be compared.

13. Diffusion Bonding Studies

A series of four tests were conducted to evaluate the diffusion bonding characteristics of eight combinations of refractory materials at temperatures of 1200° and 1400°F. The tendency of two materials to diffusion bond under certain conditions of temperature and pressure is an important consideration in the selection of materials of construction for isolation valves that are required to operate after long time exposures at elevated temperature.

A simple compression test rig, which utilizes the initial torque load plus the difference in thermal expansion coefficients between a molybdenum tie-bolt and the washer-like test specimens to obtain the desired test pressure, was used in these tests. A schematic of the test rig, including the location of the various test materials, is shown in Figure 32. Pertinent data with respect to the test materials are given in Table XII. Prior to the final assembly of the test specimens on the molybdenum tie-bolt, the surfaces of each specimen were lightly polished with 400-A Durite (Silicon Carbide) abrasive paper, cleaned in acetone and rinsed in ethyl alcohol. After assembly, the tie-bolts of all the assemblies were torqued to 15 ft-lbs. The calculated initial compressive stress was approximately 35,000 psi at a test temperature of 1200°F.

Of the four assemblies, one was wrapped in tantalum foil and heated for 100 hours in a vacuum at 1200° $\pm$ 15°F. At the start of the test the pressure was 1.4×10^{-4} torr and subsequently decreased to a value of 9×10^{-6} torr at the time the test was terminated. Two of the remaining three assemblies of the type illustrated in Figure 33, were sealed in 3/4-inch Schedule 80 x 5-inch long Cb-1Zr alloy capsules. A fourth assembly of the type illustrated in Figure 34 was sealed in a 3/4-inch Schedule 80 x 3.5-inch long Cb-1Zr capsule. All three capsules contained approximately 10 grams of potassium which had been slagged, filtered, distilled and hot trapped. The potassium was transferred direct from the hot trap to the capsule under vacuum, and the capsule was subsequently sealed using electron beam welding techniques. The

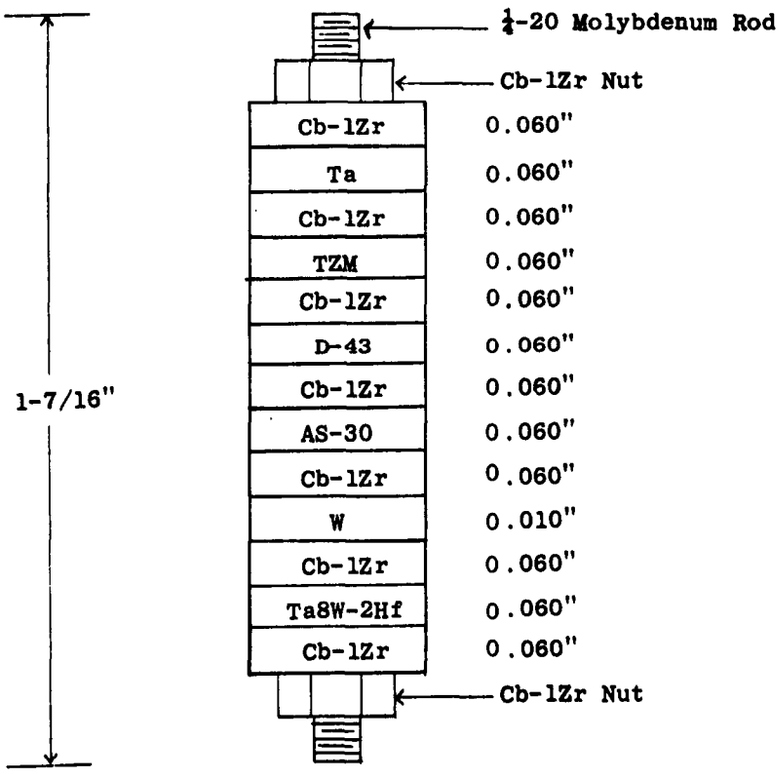

Figure 32. Schematic of Diffusion Bonding Test Assembly Illustrating the Location of the Test Materials.

TABLE XII

SPECIMEN MATERIALS USED IN BONDING STUDIES

Material	Nominal Composition	Condition	Heat No.	Thickness Inch
Cb-1Zr	1% Zr, Bal. Cb	2200°F-1 Hour, Recrystallized	519(1)	0.060
Ta	Unalloyed Ta	2200°F-1 Hour, Recrystallized	1031590(2)	0.060
T-111	8% W, 2% Hf, Bal. Ta	2800°F-1½ Hours, Recrystallized	2511(3)	0.060
W	Unalloyed W	1850°F-1 Hour, Stress-Relieved	U45-2945(4)	0.010
TZM	0.5% Ti, 0.08% Zr, Bal. Mo	2300°F-1 Hour, Stress-Relieved	KDTZM971B(5)	0.060
AS-30	20% W, 1% Zr, 0.08% C, Bal. Cb	1900°F-1 Hour, Stress-Relieved	S-182(6)	0.050
D-43	10% W, 1% Zr, 0.1% C, Bal. Cb	2200°F-1 Hour, Stress-Relieved	322(7)	0.060
Alumina	99.7% Al_2O_3		----(8)	0.25

NOTE:

Vendor:
(1) Stellite Division, UCC
(2) Kawecki Chemical Company
(3) National Research Corporation
(4) General Electric Company
(5) Universal Cyclops Steel Corporation
(6) General Electric Research Laboratory
(7) DuPont
(8) Coors Porcelain Company

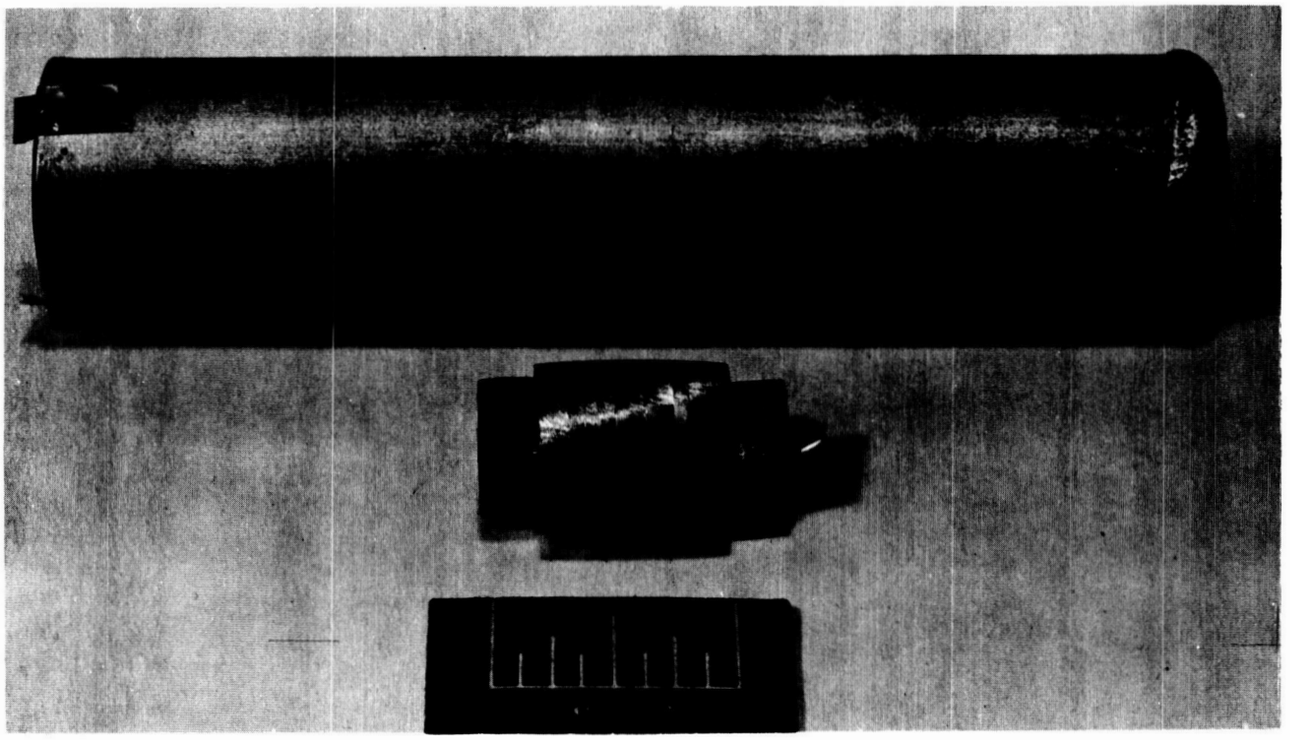

Figure 33. Test Assembly #2 with Cb-1Zr Alloy Capsule Prior to Filling with Potassium and Sealing Under Vacuum. (C63112941)

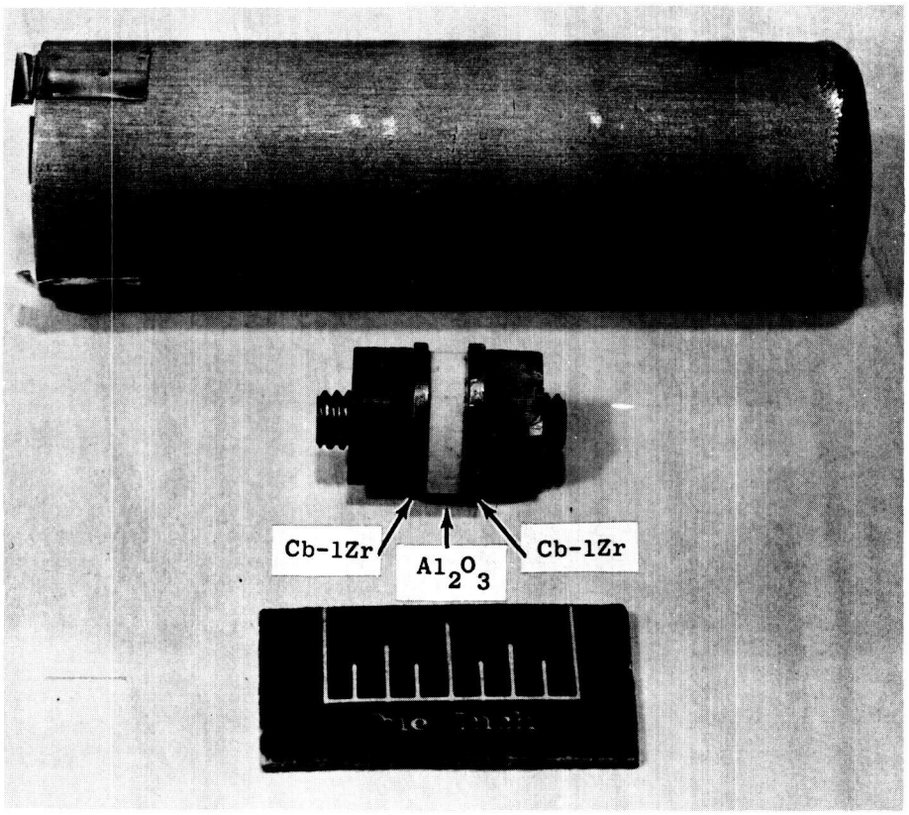

Figure 34. Test Assembly #5 with Cb-1Zr Alloy Capsule Prior to Filling with Potassium and Sealing Under Vacuum. (C63112940)

oxygen content of the potassium purified and transferred in the manner described above is less than 50 ppm as analyzed by mercury amalgamation techniques. One capsule was exposed in a vacuum for 100 hours at 1200° ± 15°F and two capsules were exposed for 100 hours at 1400° ± 15°F. The initial vacuums achieved in the 1200°F test and the two 1400°F tests were 1-2 x 10^{-5} torr and the vacuums at termination of the test were 1-2 x 10^{-6} torr.

After the test exposures, the two test assemblies that were exposed to vacuum and potassium at 1200°F were removed from the test assembly, and the Cb-1Zr alloy nuts were loosened but not removed as shown in Figures 35 and 36. Visual observations of apparent bonding between the various material combinations with Cb-1Zr alloy were made and the results are recorded in Tables XIII and XIV. The test rig then was completely disassembled and the specimens were mounted in cold setting plastic in preparation for metallographic examination. Each group of specimens that exhibited a tendency to bond was placed in a single mount, and an attempt was made to maintain the integrity of the bond. However, the bonds were so weak that all the specimens separated in the initial stages of preparing the metallographic mount. The most encouraging data, from the standpoint of the operation of valves in liquid potassium, is the fact that the Cb-1Zr alloy nuts were removed from the molybdenum tie-bolts with little difficulty. The calculated contact stress of the threads with the system at temperature was approximately 115,000 psi.

The results of the metallographic examination are summarized in Tables XIII and XIV. Generally, with the exception of "point bonding", there is little or no evidence of any interdiffusion between the seven material combinations. An example of material pull-out due to point bonding can be seen on the surface of the Cb-1Zr alloy in Figure 37. The only other observation that was made was in the varying etching characteristics of the surface of the Cb-1Zr alloy that was in contact with Mo-TZM alloy (both in vacuum and liquid potassium environment) and D-43 alloy (in vacuum environment only) to a depth of 0.001 to 0.003 inches. Although it is possible that this effect is the result of carbon diffusion across the interface, other factors such as varying amounts of cold work and residual atmospheric contamination from the processing operations could be also responsible for the slightly different etching characteristics of the surface. Microhardness traverses across the specimens from the interfaces to the centers revealed no appreciable hardening of either material of the Cb-1Zr/Mo-TZM or Cb-1Zr/D-43 couples.

From the preliminary data from this investigation, Mo-TZM was selected as the material for the valve plugs to be used in the fabrication of valves for Loop II and the Pre-prototype Loop.

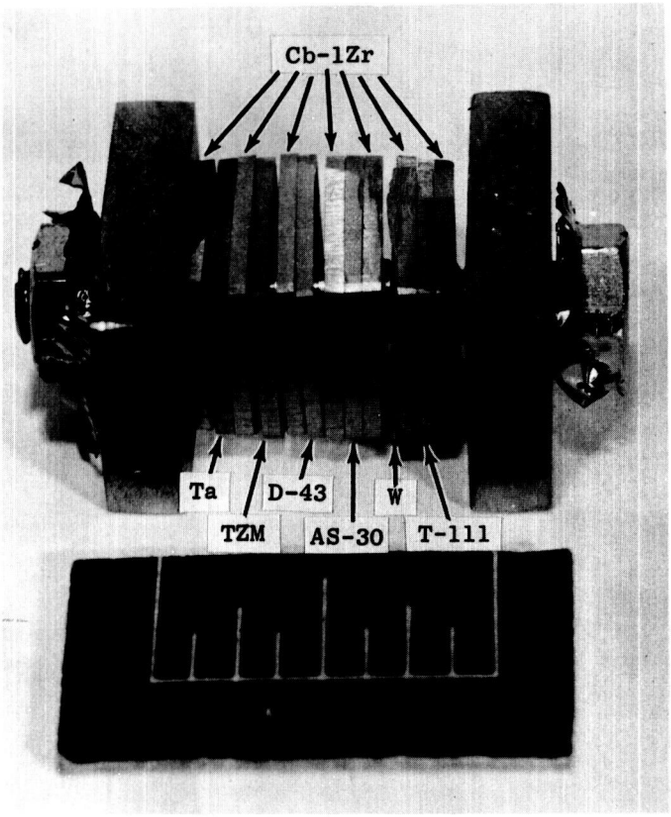

Figure 35. Test Assembly #1 After Vacuum Exposure for 100 Hours at 1200°F, Followed by Loosening of End Bolts. Note Bonding Between Specimens. (C63091204)

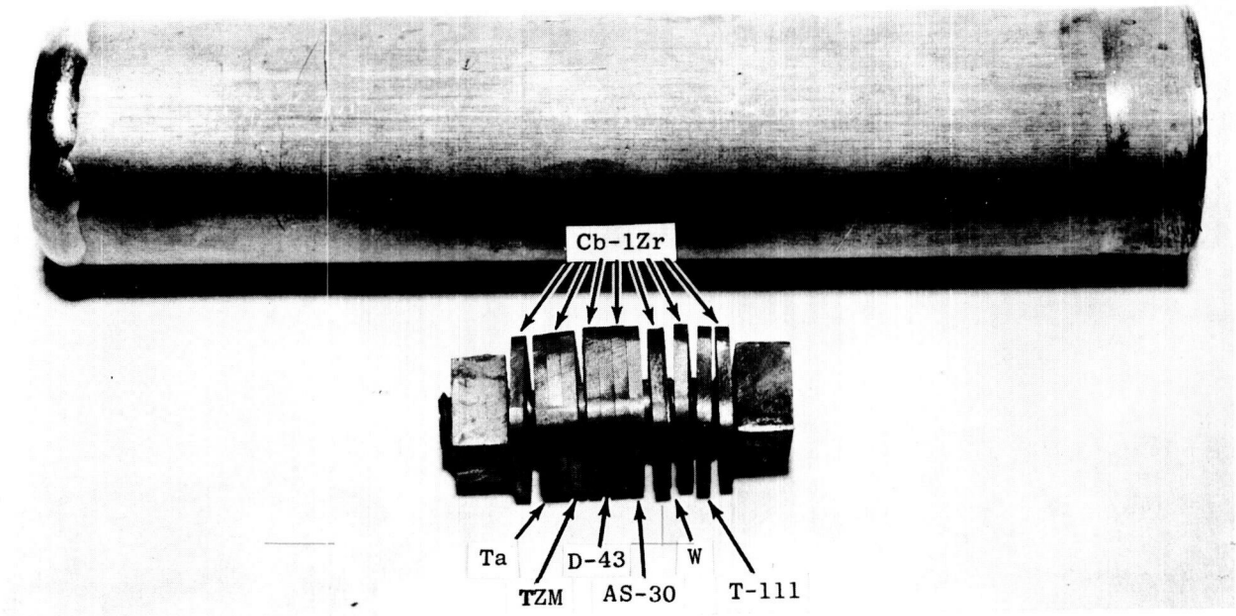

Figure 36. Test Assembly #2 After Exposure to Potassium for 100 Hours at 1200°F Inside of Cb-1Zr Capsule, Followed by Loosening of End Bolts. Note Bonding. (C63102105)

TABLE XIII

SUMMARY OF METALLOGRAPHIC OBSERVATIONS OF DIFFUSION BONDING TEST SPECIMENS ENCLOSED IN TANTALUM AND EXPOSED TO A VACUUM FOR 100 HOURS AT 1200°F (ASSEMBLY #1)

	Metallographic Observations
* Cb-1Zr (End Plate)	
----------	Slight Point Bonding/Pull-Out
Cb-1Zr	
----------	Slight Indication of Point Bonding/Pull-Out
Ta	
----------	No Bonding Effect Observed
Cb-1Zr	
----------	No Bonding Effect Observed
TZM	
----------	Etching Effect at Surface of Cb-1Zr
Cb-1Zr	
----------	Etching Effect at Surface of Cb-1Zr
D-43	
----------	No Bonding Effect Observed
Cb-1Zr	
----------	No Bonding Effect Observed
AS-30	
----------	No Bonding Effect Observed
Cb-1Zr	
----------	Slight Point Bonding/Pull-Out; Delamination of W
W	
----------	Slight Point Bonding/Pull-Out; Delamination of W
Cb-1Zr	
----------	Slight Indication of Point Bonding/Pull-Out
Ta-8W-2Hf	
----------	Slight Indication of Mechanical Bond
Cb-1Zr	

* Bonding was visually observed on the sample within each of these groups at the termination of the test and prior to mounting for metallographic examination.

TABLE XIV

SUMMARY OF METALLOGRAPHIC OBSERVATIONS OF DIFFUSION BONDING TEST SPECIMENS EXPOSED TO POTASSIUM FOR 100 HOURS AT 1200°F (ASSEMBLY #2)

	Metallographic Observations
* Cb-1Zr (End Plate)	
----------	Slight Point Bonding/Pull-Out
Cb-1Zr	
----------	No Bonding Effect Observed
Ta	
----------	Slight Point Bonding/Pull-Out
Cb-1Zr	
----------	Etching Effect at Surface of Cb-1Zr
TZM	
----------	Etching Effect at Surface of Cb-1Zr
Cb-1Zr	
----------	Slight Mechanical Bond
D-43	
----------	No Bonding Effect Observed
Cb-1Zr	
----------	Slight Indication of Point Bonding/Pull-Out
AS-30	
----------	No Bonding Effect Observed
Cb-1Zr	
----------	Slight Bonding; Delamination of W
W	
----------	Slight Bonding/Area Pull-Out; Delamination of W
Cb-1Zr	
----------	Slight Point Bonding Pull-Out
Ta-8W-2Hf	
----------	No Bonding Effect Observed
Cb-1Zr (End Plate)	

* Bonding was observed on samples within each of these groups at the termination of the test and prior to mounting for metallographic examination.

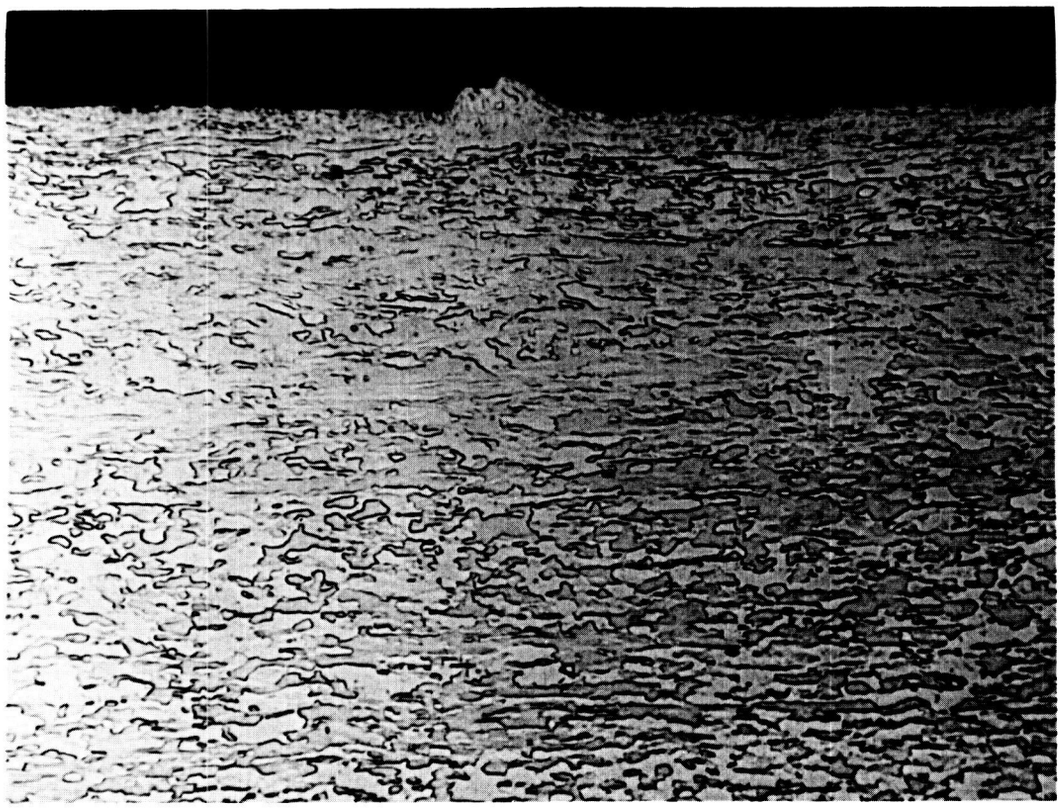

Figure 37. Surface of Cb-1Zr Alloy Specimen After 100 Hours in Contact with Cb-1Zr Alloy at 1200°F in Vacuum. Note Point Bonding and Subsequent Pull-Out.

Etchant: 60%Glycerine-20%HF-20%HNO_3 Mag: 500X (K380)

III FUTURE WORK

A. The evaluation of Loop I will be completed during the next quarter.

B. Fabrication of Loop II will be concluded and test operation started.

C. The design of the Pre-prototype Loop will be finished and the drawings submitted for approval.

D. The two EM pumps for the Pre-prototype Loop will be fabricated during the next quarter. Two sets of parts of the fast response pressure transducer will be machined. Assembly of this component will be delayed pending the observation of the performance of one of these units in Loop II. Filling of the six Taylor pressure transducers with high-purity NaK will be completed.

E. Qualification tests on the chamber for the Pre-prototype Loop will be completed by the vendor.

F. The Mo-TZM alloy — Cb-1Zr refluxing capsule tests will be prepared and put in operation near the end of the next quarter.

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